



A11103 811222

NIST
PUBLICATIONSNIST SPECIAL PUBLICATION **260-119**

U.S. DEPARTMENT OF COMMERCE/Technology Administration/National Institute of Standards and Technology

*Standard Reference Materials:***Antireflecting-Chromium
Linewidth Standard, SRM 473,
for Calibration of Optical Microscope
Linewidth Measuring Systems**QC
100
.U57
260-
119
1992**Carol F. Vezzetti, Ruth N. Varner, and James E. Potzick**

The National Institute of Standards and Technology was established in 1988 by Congress to "assist industry in the development of technology . . . needed to improve product quality, to modernize manufacturing processes, to ensure product reliability . . . and to facilitate rapid commercialization . . . of products based on new scientific discoveries."

NIST, originally founded as the National Bureau of Standards in 1901, works to strengthen U.S. industry's competitiveness; advance science and engineering; and improve public health, safety, and the environment. One of the agency's basic functions is to develop, maintain, and retain custody of the national standards of measurement, and provide the means and methods for comparing standards used in science, engineering, manufacturing, commerce, industry, and education with the standards adopted or recognized by the Federal Government.

As an agency of the U.S. Commerce Department's Technology Administration, NIST conducts basic and applied research in the physical sciences and engineering and performs related services. The Institute does generic and precompetitive work on new and advanced technologies. NIST's research facilities are located at Gaithersburg, MD 20899, and at Boulder, CO 80303. Major technical operating units and their principal activities are listed below. For more information contact the Public Inquiries Desk, 301-975-3058.

Technology Services

- Manufacturing Technology Centers Program
- Standards Services
- Technology Commercialization
- Measurement Services
- Technology Evaluation and Assessment
- Information Services

Electronics and Electrical Engineering Laboratory

- Microelectronics
- Law Enforcement Standards
- Electricity
- Semiconductor Electronics
- Electromagnetic Fields¹
- Electromagnetic Technology¹

Chemical Science and Technology Laboratory

- Biotechnology
- Chemical Engineering¹
- Chemical Kinetics and Thermodynamics
- Inorganic Analytical Research
- Organic Analytical Research
- Process Measurements
- Surface and Microanalysis Science
- Thermophysics²

Physics Laboratory

- Electron and Optical Physics
- Atomic Physics
- Molecular Physics
- Radiometric Physics
- Quantum Metrology
- Ionizing Radiation
- Time and Frequency¹
- Quantum Physics¹

Manufacturing Engineering Laboratory

- Precision Engineering
- Automated Production Technology
- Robot Systems
- Factory Automation
- Fabrication Technology

Materials Science and Engineering Laboratory

- Intelligent Processing of Materials
- Ceramics
- Materials Reliability¹
- Polymers
- Metallurgy
- Reactor Radiation

Building and Fire Research Laboratory

- Structures
- Building Materials
- Building Environment
- Fire Science and Engineering
- Fire Measurement and Research

Computer Systems Laboratory

- Information Systems Engineering
- Systems and Software Technology
- Computer Security
- Systems and Network Architecture
- Advanced Systems

Computing and Applied Mathematics Laboratory

- Applied and Computational Mathematics²
- Statistical Engineering²
- Scientific Computing Environments²
- Computer Services²
- Computer Systems and Communications²
- Information Systems

¹At Boulder, CO 80303.

²Some elements at Boulder, CO 80303.

NIST Special Publication 260-119

Standard Reference Materials:

Antireflecting-Chromium Linewidth Standard, SRM 473, for Calibration of Optical Microscope Linewidth Measuring Systems

Carol F. Vezzetti
Ruth N. Varner
James E. Potzick

Precision Engineering Division
Manufacturing Engineering Laboratory
National Institute of Standards and Technology
Gaithersburg, MD 20899



U.S. DEPARTMENT OF COMMERCE, Barbara Hackman Franklin, Secretary
TECHNOLOGY ADMINISTRATION, Robert M. White, Under Secretary for Technology
NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY, John W. Lyons, Director

Issued September 1992

National Institute of Standards and Technology Special Publication 260-119
Natl. Inst. Stand. Technol. Spec. Publ. 260-119, 48 pages (Sept. 1992)
CODEN: NSPUE2

U.S. GOVERNMENT PRINTING OFFICE
WASHINGTON: 1992

For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402-9325

Preface

Standard Reference Materials (SRM's) as defined by the National Institute of Standards and Technology (NIST) are well-characterized materials, produced in quantity and certified for one or more physical or chemical properties. They are used to assure the accuracy and compatibility of measurements throughout the Nation. SRM's are widely used as primary standards in many diverse fields in science, industry, and technology, both within the United States and throughout the world. They are also used extensively in the fields of environmental and clinical analysis. In many applications, traceability of quality control and measurement processes to the national measurement system is carried out through the mechanism and use of SRM's. For many of the Nation's scientists and technologists, it is therefore of more than passing interest to know the details of the measurements made at NIST in arriving at the certified values of the SRM's produced. The NIST Special Publication 260 Series is a series of papers reserved for this purpose.

The 260 Series is dedicated to the dissemination of information on different phases of the preparation, measurement, certification, and use of NIST SRM's. In general, much more detail will be found in these papers than is generally allowed, or desirable, in scientific journal articles. This enables the user to assess the validity and accuracy of the measurement processes employed, to judge the statistical analysis, and to learn details of techniques and methods utilized for work entailing greatest care and accuracy. These papers also should provide sufficient additional information so SRM's can be utilized in new applications in diverse fields not foreseen at the time the SRM was originally issued.

Inquiries concerning the technical content of this paper should be directed to the author(s). Other questions concerned with the availability, delivery, price, and so forth, will receive prompt attention from:

• Standard Reference Materials Program
Bldg. 202, Rm. 204
National Institute of Standards and Technology
Gaithersburg, MD 20899

William P. Reed, Chief
Standard Reference Materials Program

OTHER NIST PUBLICATIONS IN THIS SERIES

- McKenzie, R. L., ed., NIST Standard Reference Materials Catalog 1990-91, NIST Spec. Publ. 260 (January 1990)
- Michaelis, R. E., and Wyman, L. L., Standard Reference Materials: Preparation of White Cast Iron Spectrochemical Standards, NBS Misc. Publ. 260-1 (June 1964). COM74-11061**
- Michaelis, R. E., Wyman, L. L., and Flitsch, R., Standard Reference Materials: Preparation of NBS Copper-Base Spectrochemical Standards, NBS Misc. Publ. 260-2 (October 1964). COM74-11063**
- Michaelis, R. E., Yakowitz, H., and Moore, G. A., Standard Reference Materials: Metallographic Characterization of an NBS Spectrometric Low-Alloy Steel Standard, NBS Misc. Publ. 260-3 (October 1964). COM74-11060**
- Alvarez, R., and Flitsch, R., Standard Reference Materials: Accuracy of Solution X-Ray Spectrometric Analysis of Copper-Base Alloys, NBS Misc. Publ. 260-5 (March 1965). PB168068**
- Shultz, J. I., Standard Reference Materials: Methods for the Chemical Analysis of White Cast Iron Standards, NBS Misc. Publ. 260-6 (July 1965). COM74-11068**
- Bell, R. K., Standard Reference Materials: Methods for the Chemical Analysis of NBS Copper-Base Spectrochemical Standards, NBS Misc. Publ. 260-7 (October 1965). COM74-11067**
- Richmond, M. S., Standard Reference Materials: Analysis of Uranium Concentrates at the National Bureau of Standards, NBS Misc. Publ. 260-8 (December 1965). COM74-11066**
- Anspach, S. C., Cavallo, L. M., Garfinkel, S. B., Hutchinson, J. M. R., and Smith, C. N., Standard Reference Materials: Half Lives of Materials Used in the Preparation of Standard Reference Materials of Nineteen Radioactive Nuclides Issued by the National Bureau of Standards, NBS Misc. Publ. 260-9 (November 1965). COM74-11065**
- Yakowitz, H., Vieth, D. L., Heinrich, K. F. J., and Michaelis, R. E., Standard Reference Materials: Homogeneity Characterization of NBS Spectrometric Standards II: Cartridge Brass and Low-Alloy Steel, NBS Misc. Publ. 260-10 (December 1965). COM74-11064**
- Napolitano, A., and Hawkins, E. G., Standard Reference Materials: Viscosity of Standard Lead-Silica Glass, NBS Misc. Publ. 260-11 (November 1966).
- Yakowitz, H., Vieth, D. L., and Michaelis, R. E., Standard Reference Materials: Homogeneity Characterization of NBS Spectrometric Standards III: White Cast Iron and Stainless Steel Powder Compact, NBS Misc. Publ. 260-12 (September 1966).
- Menis, O., and Sterling, J. T., Standard Reference Materials: Determination of Oxygen in Ferrous Materials—SRM 1090, 1091, and 1092, NBS Misc. Publ. 260-14 (September 1966).
- Yakowitz, H., Michaelis, R. E., and Vieth, D. L., Standard Reference Materials: Homogeneity Characterization of NBS Spectrometric Standards IV: Preparation and Microprobe Characterization of W-20% Mo Alloy Fabricated by Powder Metallurgical Methods, NBS Spec. Publ. 260-16 (January 1969). COM74-11062**
- Paule, R. C., and Mandel, J., Standard Reference Materials: Analysis of Interlaboratory Measurements on the Vapor Pressure of Gold (Certification of Standard Reference Material 745). NBS Spec. Publ. 260-19 (January 1970). PB190071**
- Paule, R. C., and Mandel, J., Standard Reference Materials: Analysis of Interlaboratory Measurements on the Vapor Pressures of Cadmium and Silver, NBS Spec. Publ. 260-21 (January 1971). COM74-11359**
- Yakowitz, H., Fiori, C. E., and Michaelis, R. E., Standard Reference Materials: Homogeneity Characterization of Fe-3 Si Alloy, NBS Spec. Publ. 260-22 (February 1971). COM74-11357**
- Napolitano, A., and Hawkins, E. G., Standard Reference Materials: Viscosity of a Standard Borosilicate Glass, NBS Spec. Publ. 260-23 (December 1970). COM71-00157**
- Sappenfield, K. M., Marinenko, G., and Hague, J. L., Standard Reference Materials: Comparison of Redox Standards, NBS Spec. Publ. 260-24 (January 1972). COM72-50058**
- Hicho, G. E., Yakowitz, H., Rasberry, S. D., and Michaelis, R. E., Standard Reference Materials: A Standard Reference Material Containing Nominally Four Percent Austenite, NBS Spec. Publ. 260-25 (February 1971). COM74-11356**

- Martin, J. F., Standard Reference Materials: National Bureau of Standards-US Steel Corporation Joint Program for Determining Oxygen and Nitrogen in Steel, NBS Spec. Publ. 260-26 (February 1971). PB 81176620**
- Garner, E. L., Machlan, L. A., and Shields, W. R., Standard Reference Materials: Uranium Isotopic Standard Reference Materials, NBS Spec. Publ. 260-27 (April 1971). COM74-11358**
- Heinrich, K. F. J., Myklebust, R. L., Rasberry, S. D., and Michaelis, R. E., Standard Reference Materials: Preparation and Evaluation of SRM's 481 and 482 Gold-Silver and Gold-Copper Alloys for Microanalysis, NBS Spec. Publ. 260-28 (August 1971). COM71-50365**
- Geller, S. B., Standard Reference Materials: Calibration of NBS Secondary Standard Magnetic Tape (Computer Amplitude Reference) Using the Reference Tape Amplitude Measurement "Process A-Model 2," NBS Spec. Publ. 260-29 (June 1971). COM71-50282**
- Gorozhanina, R. S., Freedman, A. Y., and Shaievitch, A. B. (translated by M. C. Selby), Standard Reference Materials: Standard Samples Issued in the USSR (A Translation from the Russian), NBS Spec. Publ. 260-30 (June 1971). COM71-50283**
- Hust, J. G., and Sparks, L. L., Standard Reference Materials: Thermal Conductivity of Electrolytic Iron SRM 734 from 4 to 300 K, NBS Spec. Publ. 260-31 (November 1971). COM71-50563**
- Mavrodineanu, R., and Lazar, J. W., Standard Reference Materials: Standard Quartz Cuvettes for High Accuracy Spectrophotometry, NBS Spec. Publ. 260-32 (December 1973). COM74-50018**
- Wagner, H. L., Standard Reference Materials: Comparison of Original and Supplemental SRM 705, Narrow Molecular Weight Distribution Polystyrene, NBS Spec. Publ. 260-33 (May 1972). COM72-50526**
- Sparks, L. L., and Hust, J. G., Standard Reference Materials: Thermal Conductivity of Austenitic Stainless Steel, SRM 735 from 5 to 280 K, NBS Spec. Publ. 260-35 (April 1972.) COM72-50368**
- Cali, J. P., Mandel, J., Moore, L. J., and Young, D. S., Standard Reference Materials: A Referee Method for the Determination of Calcium in Serum NBS SRM 915, NBS Spec. Publ. 260-36 (May 1972). COM72-50527**
- Shultz, J. I., Bell, R. K., Rains, T. C., and Menis, O., Standard Reference Materials: Methods of Analysis of NBS Clay Standards, NBS Spec. Publ. 260-37 (June 1972). COM72-50692**
- Clark, A. F., Denson, V. A., Hust, J. G., and Powell, R. L., Standard Reference Materials: The Eddy Current Decay Method for Resistivity Characterization of High-Purity Metals, NBS Spec. Publ. 260-39 (May 1972). COM72-50529**
- McAdie, H. G., Garn, P. D., and Menis, O., Standard Reference Materials: Selection of Thermal Analysis Temperature Standards Through a Cooperative Study (SRM 758, 759, 760), NBS Spec. Publ. 260-40 (August 1972) COM72-50776**
- Wagner, H. L., and Verdier, P. H., eds., Standard Reference Materials: The Characterization of Linear Polyethylene, SRM 1475, NBS Spec. Publ. 260-42 (September 1972). COM72-50944**
- Yakowitz, H., Ruff, A. W., and Michaelis, R. E., Standard Reference Materials: Preparation and Homogeneity Characterization of an Austenitic Iron-Chromium-Nickel Alloy, NBS Spec. Publ. 260-43 (November 1972). COM73-50760**
- Schooley, J. F., Soulen, R. J., Jr., and Evans, G. A., Jr., Standard Reference Materials: Preparation and Use of Superconductive Fixed Point Devices, SRM 767, NBS Spec. Publ. 260-44 (December 1972). COM73-50037**
- Greifer, B., Maienthal, E. J., Rains, T. C., and Rasberry, S. D., Standard Reference Materials: Powdered Lead-Based Paint, SRM 1579, NBS Spec. Publ. 260-45 (March 1973). COM73-50226**
- Hust, J. G., and Giarratano, P. J., Standard Reference Materials: Thermal Conductivity and Electrical Resistivity Standard Reference Materials: Austenitic Stainless Steel, SRM's 735 and 798, from 4 to 1200 K, NBS Spec. Publ. 260-46 (March 1975). COM75-10339**
- Hust, J. G., Standard Reference Materials: Electrical Resistivity of Electrolytic Iron, SRM 797, and Austenitic Stainless Steel, SRM 798, from 5 to 280 K, NBS Spec. Publ. 260-47 (February 1974). COM74-50176**
- Mangum, B. W., and Wise, J. A., Standard Reference Materials: Description and Use of Precision Thermometers for the Clinical Laboratory, SRM 933 and SRM 934, NBS Spec. Publ. 260-48 (May 1974). Superseded by NIST Spec. Publ. 260-113.
- Carpenter, B. S., and Reimer, G. M., Standard Reference Materials: Calibrated Glass Standards for Fission Track Use, NBS Spec. Publ. 260-49 (November 1974). COM74-51185**

- Hust, J. G., and Giarratano, P. J., Standard Reference Materials: Thermal Conductivity and Electrical Resistivity Standard Reference Materials: Electrolytic Iron, SRM's 734 and 797 from 4 to 1000 K, NBS Spec. Publ. 260-50 (June 1975). COM75-10698**
- Mavrodineanu, R., and Baldwin, J. R., Standard Reference Materials: Glass Filters As a Standard Reference Material for Spectrophotometry-Selection, Preparation, Certification, Use-SRM 930 NBS Spec. Publ. 260-51 (November 1975). COM75-10339**
- Hust, J. G., and Giarratano, P. J., Standard Reference Materials: Thermal Conductivity and Electrical Resistivity Standard Reference Materials 730 and 799, from 4 to 3000 K, NBS Spec. Publ. 260-52 (September 1975). COM75-11193**
- Durst, R. A., Standard Reference Materials: Standardization of pH Measurements, NBS Spec. Publ. 260-53 (February 1988, Revision of December 1975 version).
- Burke, R. W., and Mavrodineanu, R., Standard Reference Materials: Certification and Use of Acidic Potassium Dichromate Solutions as an Ultraviolet Absorbance Standard, NBS Spec. Publ. 260-54 (August 1977). PB272168**
- Ditmars, D. A., Cezairliyan, A., Ishihara, S., and Douglas, T. B., Standard Reference Materials: Enthalpy and Heat Capacity; Molybdenum SRM 781, from 273 to 2800 K, NBS Spec. Publ. 260-55 (September 1977). PB272127**
- Powell, R. L., Sparks, L. L., and Hust, J. G., Standard Reference Materials: Standard Thermocouple Material, Pt-67: SRM 1967, NBS Spec. Publ. 260-56 (February 1978). PB277172**
- Barnes, J. D., and Martin, G. M., Standard Reference Materials: Polyester Film for Oxygen Gas Transmission Measurements SRM 1470, NBS Spec. Publ. 260-58 (June 1979). PB297098**
- Velapoldi, R. A., Paule, R. C., Schaffer, R., Mandel, J., and Moody, J. R., Standard Reference Materials: A Reference Method for the Determination of Sodium in Serum, NBS Spec. Publ. 260-60 (August 1978). PB286944**
- Verdier, P. H., and Wagner, H. L., Standard Reference Materials: The Characterization of Linear Polyethylene (SRM 1482, 1483, 1484), NBS Spec. Publ. 260-61 (December 1978). PB289899**
- Soulen, R. J., and Dove, R. B., Standard Reference Materials: Temperature Reference Standard for Use Below 0.5 K (SRM 768), NBS Spec. Publ. 260-62 (April 1979). PB294245**
- Velapoldi, R. A., Paule, R. C., Schaffer, R., Mandel, J., Machlan, L. A., and Gramlich, J. W., Standard Reference Materials: A Reference Method for the Determination of Potassium in Serum, NBS Spec. Publ. 260-63 (May 1979). PB297207**
- Velapoldi, R. A., and Mielenz, K. D., Standard Reference Materials: A Fluorescence Standard Reference Material Quinine Sulfate Dihydrate (SRM 936), NBS Spec. Publ. 260-64 (January 1980). PB80-132046**
- Marinenko, R. B., Heinrich, K. F. J., and Ruegg, F. C., Standard Reference Materials: Micro-Homogeneity Studies of NBS Standard Reference Materials, NBS Research Materials, and Other Related Samples, NBS Spec. Publ. 260-65 (September 1979). PB300461**
- Venable, W. H., Jr., and Eckerle, K. L., Standard Reference Materials: Didymium Glass Filters for Calibrating the Wavelength Scale of Spectrophotometers-SRM 2009, 2010, 2013, and 2014, NBS Spec. Publ. 260-66 (October 1979). PB80-104961**
- Velapoldi, R. A., Paule, R. C., Schaffer, R., Mandel, J., Murphy, T. J., and Gramlich, J. W., Standard Reference Materials: A Reference Method for the Determination of Chloride in Serum, NBS Spec. Publ. 260-67 (November 1979). PB80-110117**
- Mavrodineanu, R., and Baldwin, J. R., Standard Reference Materials: Metal-On-Quartz Filters as a Standard Reference Material for Spectrophotometry SRM 2031, NBS Spec. Publ. 260-68 (April 1980). PB80-197486**
- Velapoldi, R. A., Paule, R. C., Schaffer, R., Mandel, J., Machlan, L. A., Garner, E. L., and Rains, T. C., Standard Reference Materials: A Reference Method for the Determination of Lithium in Serum, NBS Spec. Publ. 260-69 (July 1980). PB80-209117**
- Marinenko, R. B., Biancaniello, F., Boyer, P. A., Ruff, A. W., and DeRobertis, L., Standard Reference Materials: Preparation and Characterization of an Iron-Chromium-Nickel Alloy for Microanalysis, NBS Spec. Publ. 260-70 (May 1981). PB84-165349**
- Seward, R. W., and Mavrodineanu, R., Standard Reference Materials: Summary of the Clinical Laboratory Standards Issued by the National Bureau of Standards, NBS Spec. Publ. 260-71 (November 1981). PB82-135161**
- Reeder, D. J., Coxon, B., Enagonio, D., Christensen, R. G., Schaffer, R., Howell, B. F., Paule, R. C., and Mandel, J., Standard Reference Materials: SRM 900, Antiepilepsy Drug Level Assay Standard, NBS Spec. Publ. 260-72 (June 1981). PB81-220758

- Interrante, C. G., and Hicho, G. E., Standard Reference Materials: A Standard Reference Material Containing Nominally Fifteen Percent Austenite (SRM 486), NBS Spec. Publ. 260-73 (January 1982). PB82-215559**
- Marinenko, R. B., Standard Reference Materials: Preparation and Characterization of K-411 and K-414 Mineral Glasses for Microanalysis: SRM 470, NBS Spec. Publ. 260-74 (April 1982). PB82-221300**
- Weidner, V. R., and Hsia, J. J., Standard Reference Materials: Preparation and Calibration of First Surface Aluminum Mirror Specular Reflectance Standards (SRM 2003a), NBS Spec. Publ. 260-75 (May 1982). PB82-221367**
- Hicho, G. E., and Eaton, E. E., Standard Reference Materials: A Standard Reference Material Containing Nominally Five Percent Austenite (SRM 485a), NBS Spec. Publ. 260-76 (August 1982). PB83-115568**
- Furukawa, G. T., Riddle, J. L., Bigge, W. G., and Pfeiffer, E. R., Standard Reference Materials: Application of Some Metal SRM's as Thermometric Fixed Points, NBS Spec. Publ. 260-77 (August 1982). PB83-117325**
- Hicho, G. E., and Eaton, E. E., Standard Reference Materials: Standard Reference Material Containing Nominally Thirty Percent Austenite (SRM 487), NBS Spec. Publ. 260-78 (September 1982). PB83-115576**
- Richmond, J. C., Hsia, J. J., Weidner, V. R., and Wilmering, D. B., Standard Reference Materials: Second Surface Mirror Standards of Specular Spectral Reflectance (SRM's 2023, 2024, 2025), NBS Spec. Publ. 260-79 (October 1982). PB84-203447**
- Schaffer, R., Mandel, J., Sun, T., Cohen, A., and Hertz, H. S., Standard Reference Materials: Evaluation by an ID/MS Method of the AACC Reference Method for Serum Glucose, NBS Spec. Publ. 260-80 (October 1982). PB84-216894**
- Burke, R. W., and Mavrodineanu, R., Standard Reference Materials: Accuracy in Analytical Spectrophotometry, NBS Spec. Publ. 260-81 (April 1983). PB83-214536**
- Weidner, V. R., Standard Reference Materials: White Opal Glass Diffuse Spectral Reflectance Standards for the Visible Spectrum (SRM's 2015 and 2016), NBS Spec. Publ. 260-82 (April 1983). PB83-220723**
- Bowers, G. N., Jr., Alvarez, R., Cali, J. P., Eberhardt, K. R., Reeder, D. J., Schaffer, R., and Uriano, G. A., Standard Reference Materials: The Measurement of the Catalytic (Activity) Concentration of Seven Enzymes in NBS Human Serum SRM 909, NBS Spec. Publ. 260-83 (June 1983). PB83-239509**
- Gills, T. E., Seward, R. W., Collins, R. J., and Webster, W. C., Standard Reference Materials: Sampling, Materials Handling, Processing, and Packaging of NBS Sulfur in Coal Standard Reference Materials 2682, 2683, 2684, and 2685, NBS Spec. Publ. 260-84 (August 1983). PB84-109552**
- Swyt, D. A., Standard Reference Materials: A Look at Techniques for the Dimensional Calibration of Standard Microscopic Particles, NBS Spec. Publ. 260-85 (September 1983). PB84-112648**
- Hicho, G. E., and Eaton, E. E., Standard Reference Materials: A Standard Reference Material Containing Two and One-Half Percent Austenite, SRM 488, NBS Spec. Publ. 260-86 (December 1983). PB84-143296**
- Mangum, B. W., Standard Reference Materials: SRM 1969: Rubidium Triple-Point - A Temperature Reference Standard Near 39.30 °C, NBS Spec. Publ. 260-87 (December 1983). PB84-149996**
- Gladney, E. S., Burns, C. E., Perrin, D. R., Roelandts, I., and Gills, T. E., Standard Reference Materials: 1982 Compilation of Elemental Concentration Data for NBS Biological, Geological, and Environmental Standard Reference Materials, NBS Spec. Publ. 260-88 (March 1984). PB84-218338**
- Hust, J. G., Standard Reference Materials: A Fine-Grained, Isotropic Graphite for Use as NBS Thermophysical Property RM's from 5 to 2500 K, NBS Spec. Publ. 260-89 (September 1984). PB85-112886**
- Hust, J. G., and Lankford, A. B., Standard Reference Materials: Update of Thermal Conductivity and Electrical Resistivity of Electrolytic Iron, Tungsten, and Stainless Steel, NBS Spec. Publ. 260-90 (September 1984). PB85-115814**
- Goodrich, L. F., Vecchia, D. F., Pittman, E. S., Ekin, J. W., and Clark, A. F., Standard Reference Materials: Critical Current Measurements on an NbTi Superconducting Wire Standard Reference Material, NBS Spec. Publ. 260-91 (September 1984). PB85-118594**
- Carpenter, B. S., Standard Reference Materials: Calibrated Glass Standards for Fission Track Use (Supplement to NBS Spec. Publ. 260-49), NBS Spec. Publ. 260-92 (September 1984). PB85-113025**

- Ehrstein, J.R., Standard Reference Materials: Preparation and Certification of Standard Reference Materials for Calibration of Spreading Resistance Probes, NBS Spec. Publ. 260-93 (January 1985). PB85-177921**
- Gills, T. E., Koch, W. F., Stolz, J. W., Kelly, W. R., Paulsen, P. J., Colbert, J. C., Kirklin, D. R., Pei, P.T.S., Weeks, S., Lindstrom, R. M., Fleming, R. F., Greenberg, R. R., and Paule, R. C., Standard Reference Materials: Methods and Procedures Used at the National Bureau of Standards to Certify Sulfur in Coal SRM's for Sulfur Content, Calorific Value, Ash Content, NBS Spec. Publ. 260-94 (December 1984). PB85-165900**
- Mulholland, G. W., Hartman, A. W., Hembree, G. G., Marx, E., and Lettieri, T. R., Standard Reference Materials: Development of a 1 μ m Diameter Particle Size Standard, SRM 1690, NBS Spec. Publ. 260-95 (May 1985). SN003-003-02665-4*
- Carpenter, B. S., Gramlich, J. W., Greenberg, R. R., Machlan, L. A., DeBievre, P., Eschbach, H. L., Meyer, H., Van Audenhove, J., Connolly, V. E., Trahey, N. M., and Zook, A. C., Standard Reference Materials: Uranium-235 Isotopic Abundance Standard Reference Materials for Gamma Spectrometry Measurements, NBS Spec. Publ. 260-96 (September 1986). PB87-108544**
- Mavrodineanu, R., and Gills, T. E., Standard Reference Materials: Summary of the Coal, Ore, Mineral, Rock, and Refractory Standards Issued by the National Bureau of Standards, NBS Spec. Publ. 260-97 (September 1985). SN003-003-02688-3*
- Hust, J. G., Standard Reference Materials: Glass Fiberboard SRM for Thermal Resistance, NBS Spec. Publ. 260-98 (August 1985). SN003-003-02674-3*
- Callanan, J. E., Sullivan, S. A., and Vecchia, D. F., Standard Reference Materials: Feasibility Study for the Development of Standards Using Differential Scanning Calorimetry, NBS Spec. Publ. 260-99 (August 1985). SN003-003-02675-1*
- Taylor, J. K., Standard Reference Materials: Handbook for SRM Users, NBS Spec. Publ. 260-100 (September 1985). PB86-110897**
- Mangum, B. W., Standard Reference Materials: SRM 1970, Succinonitrile Triple-Point Standard: A Temperature Reference Standard Near 58.08 °C, NBS Spec. Publ. 260-101 (March 1986). SN003-003-02722-7*
- Weidner, V. R., Mavrodineanu, R., Mielenz, K. D., Velapoldi, R. A., Eckerle, K. L., and Adams, B., Standard Reference Materials: Holmium Oxide Solution Wavelength Standard from 240 to 640 nm - SRM 2034, NBS Spec. Publ. 260-102 (July 1986). PB86-245727**
- Hust, J. G., Standard Reference Materials: Glass Fiberblanket SRM for Thermal Resistance, NBS Spec. Publ. 260-103 (September 1985). SN003-003-02687-5*
- Mavrodineanu, R., and Alvarez, R., Standard Reference Materials: Summary of the Biological and Botanical Standards Issued by the National Bureau of Standards, NBS Spec. Publ. 260-104 (November 1985). SN003-003-02704-9*
- Mavrodineanu, R., and Rasberry, S. D., Standard Reference Materials: Summary of the Environmental Research, Analysis, and Control Standards Issued by the National Bureau of Standards, NBS Spec. Publ. 260-105 (March 1986). SN003-003-02725-1*
- Koch, W. F., ed., Standard Reference Materials: Methods and Procedures Used at the National Bureau of Standards to Prepare, Analyze, and Certify SRM 2694, Simulated Rainwater, and Recommendations for Use, NBS Spec. Publ. 260-106 (July 1986). PB86-247483**
- Hartman, A. W., and McKenzie, R. L., Standard Reference Materials: SRM 1965, Microsphere Slide (10 μ m Polystyrene Spheres), NBS Spec. Publ. 260-107 (November 1988).
- Mavrodineanu, R., and Gills, T. E., Standard Reference Materials: Summary of Gas Cylinder and Permeation Tube Standard Reference Materials Issued by the National Bureau of Standards, NBS Spec. Publ. 260-108 (May 1987).
- Candela, G. A., Chandler-Horowitz, D., Novotny, D. B., Marchiando, J. F., and Belzer, B. J., Standard Reference Materials: Preparation and Certification of an Ellipsometrically Derived Thickness and Refractive Index Standard of a Silicon Dioxide Film (SRM 2530), NIST Spec. Publ. 260-109 (October 1988).
- Kirby, R. K., and Kanare, H. M., Standard Reference Materials: Portland Cement Chemical Composition Standards (Blending, Packaging, and Testing), NBS Spec. Publ. 260-110 (February 1988).
- Gladney, E. S., O'Malley, B. T., Roelandts, I., and Gills, T. E., Standard Reference Materials: Compilation of Elemental Concentration Data for NBS Clinical, Biological, Geological, and Environmental Standard Reference Materials, NBS Spec. Publ. 260-111 (November 1987).

- Marinenko, R. B., Blackburn, D. H., and Bodkin, J. B., Standard Reference Materials: Glasses for Microanalysis: SRM's 1871-1875, NIST Spec. Publ. 260-112 (February 1990).
- Mangum, B. W., and Wise, J. A., Standard Reference Materials: Description and Use of a Precision Thermometer for the Clinical Laboratory, SRM934, NIST Spec. Publ. 260-113 (June 1990).
- Vezzetti, C. F., Varner, R. N. and Potzick, J. E., Standard Reference Materials: Bright-Chromium Linewidth Standard, SRM 476, for Calibration of Optical Microscope Linewidth Measuring Systems, NIST Spec. Publ. 260-114 (January 1991).
- Williamson, M. P., Willman, N. E., and Grubb, D. S., Standard Reference Materials: Calibration of NIST Standard Reference Material 3201 for 0.5 Inch (12.65 mm) Serial Serpentine Magnetic Tape Cartridge, NIST Spec. Publ. 260-115 (February 1991).
- Mavrodineanu, R., Burke, R. W., Baldwin, J. R., Smith, M. V., and Messman, J. D., Standard Reference Materials: Glass Filters as a Standard Reference Material for Spectrophotometry and Selection, Preparation, Certification and use of SRM 930 and SRM 1930, NIST Spec. Publ. 260-116 (in prep).
- Vezzetti, C. F., Varner, R. N., and Potzick, J. E., Standard Reference Materials: Antireflecting-Chromium Linewidth Standard, SRM 475, for Calibration of Optical Microscope Linewidth Measuring Systems, NIST Spec. Publ. 260-117 (January 1992).
- Williamson, M. P., Standard Reference Materials: Calibration of NIST Standard Reference Material 3202 for 18-Track, Parallel, and 36-Track, Parallel Serpentine, 12.65 mm (0.5 in), 1491 cpmm (37871 cpi), Magnetic Tape Cartridge, NIST Spec. Publ. 260-118 (July 1992)

*Send order with remittance to Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20102. Remittance from foreign countries should include an additional one fourth of the purchase price for postage.

**May be ordered from: National Technical Information Services (NTIS), Springfield, VA 22161.

Contents

1. Introduction	1
2. Physical Characteristics of SRM 473	1
3. Using SRM 473	3
3.1 Special Precautions	3
3.2 Microscope Calibration Procedures	6
4. Calibration of SRM 473	7
4.1 The Measurement System	8
4.2 SRM Calibration Procedure	8
4.3 Specimen and Measurement Axis Alignment	9
4.4 Feature Measurement Sequence	10
4.5 Edge Location Determination	10
4.6 Precision and Accuracy of the SRM Calibration	12
Acknowledgments	14
References	15
Table	17
Figures	18
Appendix: Process Control for SRM 473 Calibrations	27
A. Introduction	27
B. Initialization of Process Parameters	27
C. Procedures for Process Control	29
D. Updating Process Parameters	30
E. Uncertainty Statement for SRM 473	33
Acknowledgments	34
References	34
Tables	35
Figures	36

ANTIREFLECTING-CHROMIUM LINEWIDTH STANDARD, SRM 473,
FOR CALIBRATION OF OPTICAL MICROSCOPE
LINEWIDTH MEASURING SYSTEMS

C. F. Vezzetti, R. N. Varner, and J. E. Potzick

*National Institute of Standards and Technology
Gaithersburg, Maryland 20899*

ABSTRACT

The precise and accurate measurement of feature dimensions on photomasks, such as those used in the production of integrated circuits, becomes increasingly difficult as the dimensions approach the wavelength of the light used to make the measurement. The undesirable effects of optical diffraction obscure the location of the feature edges. Raggedness and nonvertical walls along the edges add to the uncertainty of the measurement.

Standard Reference Material SRM 473 was developed for use in calibrating optical microscopes for measuring linewidths in the range of 0.5 to 30 μm on antireflecting-chromium photomasks. The SRM is described, and instructions on its use and precautions concerning its care and handling are given.

The NIST linewidth measuring system and the procedures used to calibrate the SRM are discussed. The algorithm used for determining the line edge location incorporates a threshold criterion derived from analysis of microscope image profiles. The profiles are predicted by computer modeling based on the theory of partial coherence. The performance of this system is monitored by measuring line features on a control photomask before and after calibrating each SRM.

KEY WORDS: accuracy; antireflecting-chromium; calibration; control charts; critical dimensions; integrated circuits; linewidth measurement; optical microscope; photomask; precision; semiconductor industry; standard reference material; statistical process control; threshold; uncertainty.

1. Introduction

The ability to measure and control critical dimensions during the production of integrated circuits is essential to the semiconductor industry. Many measuring systems claiming high precision are now commercially available for use in determining some of these critical dimensions. As part of a continuing effort to provide means for calibrating these systems, the National Institute of Standards and Technology (NIST) has developed Optical Microscope Linewidth Measurement Standard Reference Materials.

Standard Reference Material (SRM) 473 is designed for calibrating optical microscope systems to measure linewidths on antireflecting-chromium photomasks. It was produced with conventional technology by a commercial photomask manufacturing facility. In addition to isolated opaque and clear lines for linewidth calibration, this SRM contains line patterns for checking length scale, adjusting video-type micrometers, and detecting mechanical or optical nonlinearities. The design of the calibrated pattern is described in section 2.

Section 3 gives information and precautions on the care and use of this SRM to calibrate an optical linewidth measuring system. Because of the variety of linewidth measuring systems in use today, no attempt has been made to give specific instructions for each type of microscope.

To calibrate the SRM line features, a photometric microscope with lenses selected for least aberration was modified at NIST. Except for the initial positioning, aligning, and focusing of the photomask, the entire calibration process is automated. The line features are illuminated with partially coherent green light (wavelength $0.53\ \mu\text{m}$) from a filtered incandescent source. The linewidths are determined from the image profile (image intensity versus position across a feature). A considerable amount of theoretical work was conducted to establish the location on the observed image profile that corresponds to the physical edge of a feature. The quality of the feature edge geometry of a sample from each production batch of SRM photomasks is examined with a scanning electron microscope (SEM). The limit of accuracy of the calibration measurements given in the certificate data is based on this sampled edge geometry and the agreement between theoretically modeled and experimentally generated image profiles. Section 4 contains brief descriptions of the NIST linewidth calibration system and the automated calibration process as well as discussions of the line edge location algorithm, precision, and accuracy. The process control procedures used in the calibration of this SRM are discussed in some detail in the Appendix.

2. Physical Characteristics of SRM 473

SRM 473 is made from an antireflecting-chromium photoplate by conventional photolithographic techniques. The substrate is a quartz plate of a type commonly used for fabricating integrated circuit photomasks, nominally $127\ \text{x}\ 127\ \text{x}\ 2.3\ \text{mm}$ ($5.0\ \text{x}\ 5.0\ \text{x}\ 0.09\ \text{in}$). The nominal thickness of the chromium layer is 100 nm.

Figure 1 shows the overall pattern on the chromium-coated side of the standard. The three horizontal and three vertical intersecting lines help locate the basic measurement pattern which is repeated at eight locations around the center of the standard as shown in figure 2 (a magnified view of the central area of fig. 1). A pattern identification number (1 through 8) is located within each basic pattern. Only one of these eight patterns is certified. The certificate accompanying the SRM gives the number of the certified pattern. The carpet design at the center of the photomask as well as those within each basic pattern contain no calibrated features.

Figure 3 shows the details of the features in each of the eight identical patterns on the SRM. The pattern identification number can be seen in the lower right quadrant, just above the carpet design. The vertical sides of the box surrounding the basic pattern are parallel to the calibrated line features and may be used to aid in aligning the SRM features to be perpendicular to the measurement axis.

The calibrated features are arranged in six rows. Row numbers are located at the ends of each row. Each feature within rows 1 through 5 is further identified by a letter, A through L, located immediately above the feature. Thus, 1E refers to the opaque line in row 1 at position E. Row 6 contains a single multiple-line feature with every 5th line elongated and every 10th line numbered. All rows on the SRM contain a broken horizontal fiducial line which defines the measurement position on each feature.

Row 1 consists of 12 opaque lines (1A through 1L) on a clear background, and row 2 consists of 12 clear lines (2A through 2L) on an opaque background. These opaque and clear lines are used for calibrating optical microscopes used to measure linewidths of isolated lines of either or both polarities. Nominal linewidths of these features range from 0.5 to 30 μm .

Rows 3 and 4 are intended to be used for calibrating optical microscopes for making line spacing (pitch) measurements as well as for making initial length scale adjustments when calibrating linewidth measurement systems. Row 3 consists of five features (3A through 3E), each with four opaque lines. Certified values are given for the pitch of the two interior (short) lines¹ of each feature. Nominal pitches for these features range from 2.0 to 6.0 μm . Row 4 contains a series of nine opaque lines, with certified pitch values given for the six short lines (4A through 4F) only¹. The values given on the certificate are for the pitches from line 4A to lines 4B through 4F. Pitches for the other combinations of lines (e.g., 4B to 4E) can be calculated from the certificate values, giving an array of nominal pitch values from 5.0 to 70 μm .

¹The two outer lines of each pattern in row 3 and the three unlettered (long) lines in row 4 serve as "guard lines" during the photolithographic etching process to equalize proximity effects along the line edges and are not calibrated.

Row 5 consists of four multiple-line features (5A through 5D) with approximately equal line and space widths. The widths of the left interior line and central space are certified². Nominal widths range from 1.0 to 5.0 μm . These features are useful for adjusting brightness and contrast of video image-scanning instruments and setting variable-threshold systems to achieve the proper line-to-space ratio.

The calibrated feature in row 6 is a series of 33 opaque lines, nominally 1.0 μm wide with 2.0 μm center-to-center spacing; distances from line 0 to lines 1 through 31² are certified. This feature is intended to be used as a linear scale in checking for mechanical nonlinearities and optical distortions in the linewidth measurement system (e.g., the magnification as a function of position over the field of view) and for checking the resolving power of the microscope objective.

3. Using SRM 473

The following section provides information on the care and handling of the SRM photomask and gives basic instructions and precautions on its use for calibrating optical microscope systems for measuring linewidths of features on antireflecting photomasks or similar artifacts.

3.1 Special Precautions

Contamination or damage can change the measured linewidths, invalidating the NIST calibration. Particular care should be taken during use to avoid bringing the microscope objective, or any other object, into contact with the top (chromium-coated) surface of the SRM. It is recommended that users calibrate secondary standards of their own design and use these in routine calibrations while keeping the NIST standard in safe storage. If this is done, the secondary standards should be checked periodically against the NIST standard. Also, it may be advisable for the user to calibrate one or more of the uncalibrated patterns on the SRM for use in the event that the NIST calibrated pattern is destroyed.

Precautions should be taken to prevent the accumulation of airborne and other contaminants on the SRM. If cleaning becomes necessary, use only noncorrosive wetting solutions (surfactants) at room temperature.

For cleaning we recommend the following procedure:

- Soak the SRM for 15 minutes to several hours in a mild solution of commercial mask cleaner and deionized water.

²The two outer lines of each pattern in rows 5 and 6 serve as "guard lines" during the photolithographic etching process to equalize proximity effects along the line edges and are not calibrated.

- While the mask is still immersed, brush the coated side gently with a soft lens brush; stroke parallel to the calibrated line length and in one direction.
- Rinse the mask thoroughly with deionized water.
- Blow away water droplets with a stream of clean dry air or nitrogen at room temperature.

If the contamination persists, apply a few drops of undiluted mask cleaner directly on the SRM before repeating the above cleaning process.

Removing fingerprints or other greasy contamination may require rinsing the SRM with alcohol or acetone and repeating the above cleaning process.

Inappropriate use of the NIST linewidth standards can result in inaccurate calibrations and may invalidate traceability to NIST. The practices most apt to give inaccurate calibrations when using the NIST linewidth standard include:

- a. Using the linewidth standard to calibrate a measurement system that will then be used to measure linewidths on specimens with optical properties that differ significantly from those of the standard (for example, features on silicon wafers). One important requirement for accuracy is that the image profile (or diffraction pattern) of the edge have the same shape for both the standard and the user's specimens. These image profiles will not have the same shape if the optical properties of the standard and the user's specimens differ.

When calibrating optical measuring systems that use transmitted light, it is especially important that the transmittance of the chromium film on the standard and the user's specimen match at the measuring wavelength. The transmittance of SRM 473 is less than 0.2% at a wavelength of 0.53 μm . Line edge location conditions for photomasks with transmittance greater than about 0.5% may be significantly different from those of the SRM.

When calibrating optical measuring systems that use reflected light, the standard and the user's specimen should match even more closely. The more important properties to match are the complex reflection coefficient of the patterned metal layer and the substrate, the thickness of the patterned layer, and the transmittance of the patterned layer.

- b. Using the linewidth standard to calibrate a scanning electron microscope. This SRM is designed specifically for use with optical microscopes and, as no criterion has been established for edge location in an SEM image, this SRM cannot be used to calibrate an SEM for linewidth measurements. Its use in an SEM is further discouraged because the profile of the feature could change as a result of coating the SRM with an evaporated film to reduce electrical charging, of deposition of contamination during operation of the SEM, and of detachment of the chromium during cleaning to remove evaporated films or

contaminants. (The substrate of this SRM is quartz and, even when low-voltage SEM techniques are used it is next to impossible to view the SRM features in the SEM without first coating the sample).

- c. Failing to correct for scattered (or flare) light. Although the chromium pattern on SRM 473 is not highly reflective, it includes isolated features surrounded by various large clear areas and the image profiles may exhibit a moderate component of scattered light which may vary from feature-to-feature and from the user's specimen. The intensity of the scattered light should be subtracted from all measured intensity levels before determining the edge location (see sec. 4.5). This correction has been made in the calibration of SRM 473. For the SRM user, determination of I_f for each feature by using eq (4)(sec. 4.5) would be time consuming and impractical; however, when, as for this SRM, the transmittance is low (less than 0.2%), I_o' and I_f are nearly identical and the user may consider all measured I_o' intensity to be flare light. Then the correction for flare light can be implemented simply by one of the following actions: shift the intensity zero level so that $I_o' = 0$; subtract I_o' from the measured intensities; determine the threshold level as a percent of $(I_m' - I_o')$. If the user cannot make this correction, the reflectance and transmittance of the standard used for calibration should match the reflectance and transmittance of the user's specimen at the measuring wavelength.

At the present time, NIST has two other linewidth standards (475 and 476). These two SRMs have a more limited range of linewidths than SRM 473. SRM 476 is patterned with bright chromium and SRM 475 is patterned, as is SRM 473, with antireflective chromium. We recommend that the user: (1) use the SRM that most closely matches the specimens to be measured and (2) make the scattered light correction outlined above.

- d. Using the NIST linewidth standards to generate a calibration curve that is then used for features that are larger than the largest or smaller than the smallest feature on the standard. The nominal linewidth range of SRM 473 is from 0.5 to 30 μm and this SRM will not adequately calibrate a microscope outside of this range. This is especially true for extensions much below the nominal range where the calibration curve becomes nonlinear.

The user should be aware that all standards have a precision and accuracy of calibration associated with them and, to this extent, are not perfect. The calibration of a microscope using a standard has an imprecision associated with that calibration and also has an imprecision associated with the subsequent use of that calibrated microscope to measure an unknown specimen. Therefore, the accuracy of the user's measurements cannot exceed the accuracy of the standard. The uncertainty of the final measurement on the unknown specimen is a combination of the accuracy of the standard used for calibration, the precision of the calibration measurements using the standard, and the precision of the measurements of the unknown specimen.

These and other topics are discussed more fully in the references [1-14]. The need to use good measurement techniques to achieve the best results with these linewidth standards cannot be overemphasized. The user who knows more about the potential problems is more likely to make better use of the linewidth standard.

3.2 Microscope Calibration Procedures

The following procedures are recommended for using this SRM to calibrate optical microscope systems for measuring linewidths on antireflecting photomasks. It is assumed the user is familiar with the operation of the microscope system being calibrated; no attempt is made to give detailed instruction on the use of microscope systems. The steps marked with an asterisk^(*) need only be performed the first time the system is used or after any changes in the measurement system.

Procedures	Explanatory Notes
1. Set up the measurement system for dimensional measurements; use the same procedures that will be used for measuring photomasks.	Follow manufacturers instructions or consult reference [1] for recommended procedures including adjustments for Köhler illumination.
2. Locate within the microscope field-of-view the specific basic pattern on the SRM that has been calibrated by NIST.	The pattern identification number is located in the box above the carpet design in the lower right (see fig. 2). The identification number of the calibrated pattern is given on the SRM certificate.
3.* Check the resolving power of the microscope objective by focusing on row 6.	If the objective cannot resolve clearly the lines in this feature, use another objective.
4. Align the SRM so that lines are measured in a direction perpendicular to their length.	The box surrounding the basic pattern group (see fig. 3) may be used as an alignment aid.
5. Adjust the measurement system length scale to give the same reading for the spacing of appropriate line pair(s) in row(s) 3 or 4 as the NIST value.	The line pair(s) chosen should have spacing in the same range as the dimensions of the features to be measured by the user.
6.* Check for mechanical nonlinearity and/or optical distortion by measuring the spacings of the lines in row 6, and compare the results with the NIST values.	For all further measurements, use only the portion of the field of view corresponding to the location where the differences from NIST values are relatively constant or that portion of the video-display which exhibits minimum distortion.

Procedures - continued

7. Adjust system contrast, brightness (on video-type image-scanning systems) and/or threshold level until the measured widths of both the line and space of an appropriate feature in row 5 agree as closely as possible with the NIST values. Use these same settings throughout this measurement session.

NOTE: If any changes other than refocusing, repositioning, and adjusting for flare are inadvertently made during the following steps, discard the data and start again with step 5.

8. Measure and record the widths of the calibrated features in rows 1 and 2 and/or the pitches in rows 3 and 4.

9. Derive the calibration curves as described in reference [2].

Explanatory Notes - continued

The feature chosen should have widths within the range of the anticipated measurements.

Compensate for flare light during this process and for all subsequent measurements (see paragraph c., sec. 3.1).

Use the same focusing criteria throughout and make all measurements in the same direction of travel. Compensate for flare light on all photomasks (not only this standard).

These calibration curves apply only to this system/operator combination.

The system is now ready for measurement of other antireflecting-chromium photomasks or artifacts with optical properties similar to SRM 473 (low reflectance and very low transmittance) using the same threshold value and flare-light correction procedure. If the user attempts to measure artifacts with chromium layers having transmission much greater than 0.2%, it may be necessary to measure the phase angle, ϕ , and use eq (3) to determine a different edge location threshold. These procedures are beyond the scope of this report.

Repeat the complete calibration procedure on a routine periodic basis and whenever a substantial change is made in the measurement system. The time between periodic calibrations may have to be determined empirically.

4. Calibration of SRM 473

All measurements at NIST of the SRM feature dimensions were performed on the automated optical linewidth system [3] in a laboratory with temperature controlled at 21 ± 2 °C. Linewidths and pitches are determined from the optical profile data. The uncertainty of the calibrations is a combination of the uncertainties of the measurement process, of the feature edge location algorithm, and of the geometry of the physical edge of the measured features.

4.1 The Measurement System

The measured system, diagrammed in figure 4, is built around a carefully aligned optical transmission microscope mounted on a vibration isolation table. The photomask is placed on a scanning piezoelectric flexure-pivot stage with finely controlled motion in the X (scanning) and Z (focus) directions; this stage is mounted on another stage with coarse motion leadscrews in the X and Y directions to allow positioning of the desired feature in the field of view.

The photomask is illuminated from below with Köhler illumination (i.e., each point on the lamp filament evenly illuminates the entire specimen) from an incandescent source filtered at 530 nm wavelength with a coherence parameter of 2/3 (0.6 numerical aperture condenser lens and 0.9 numerical aperture objective lens). A 20 x 400 μm slit is fixed on axis in the image plane in front of a photomultiplier tube. Image magnification at the slit is 157 times, giving an effective measurement area on the photomask of 0.127 x 2.55 μm , which is centered top-to-bottom on the feature (at the fiducial line). The photomultiplier output is amplified and digitized by a 12-bit analog-to-digital converter (ADC). Stage motion in the scanning direction is measured by a laser interferometer with resolution of 125 points per micrometer. All these devices are connected via appropriate control hardware and IEEE-488 bus to a dedicated desktop digital computer.

4.2 SRM Calibration Procedure

An outline of the overall calibration procedure is charted in figure 5. Before each complete SRM calibration, selected features on a control photomask are measured and compared with control history to ensure that the system has not changed or drifted. These selected features include spacing patterns 3E, 4F, and row 6 which have been independently calibrated by the Dimensional Metrology Group at NIST [4] to provide traceability to the standard meter. Each feature on the SRM being calibrated is then measured in sequence and the sequence repeated nine times. After each SRM calibration is completed, the control photomask is measured again.

All measurements, including the control measurements, are entered into the linewidth database. After the calibration measurements are completed the database is searched to ensure that the control was measured before and after the calibration and that these two control measurements were statistically invariate. The database entries for the calibration are combined and examined statistically: the standard deviation for each feature is calculated, number of measurements checked, etc. Criteria must be met for each of these statistical factors. If necessary, more measurements can be made and added to the database.

Once all the above conditions are met, the certificate is printed and the SRM linewidth standard is released to the Standard Reference Materials Program Office for sale. All of the calibration database files for this serial number are then stored on one flexible disk along with summary data. The disk is kept for archival storage along with the printed calibration results for each measurement, a printed summary of the statistical data, and dark-field illumination micrographs of the calibrated pattern.

4.3 Specimen and Measurement Axis Alignment

The measurement axis is the axis of the interferometer laser beam (actually the geometric center of the four beams used in this interferometer) and is defined as the X-axis. The scanning axis is the axis of motion of the piezoelectric scanning stage, and the specimen axis is a line on the surface of the photomask perpendicular to the length of the linewidth feature being measured. Ideally these axes would coincide, but in practice it is not possible to locate these axes with great precision.

In this application the perpendicular distance between parallel lines (the right edge and left edge of a feature) is measured. It is important to align the specimen axis with the measurement axis, but slight misalignment of the scanning axis causes no error because only the component of motion parallel to the measurement axis is measured and this is also the component parallel to the specimen axis. In other words, a scanning axis misalignment will cause the width measurement to "slide" slightly along the length of the line, but it will always be parallel to the line's width. Even so the scanning axis is aligned as carefully as possible, first by aligning the leadscrew stage by moving it back and forth in the Y direction and adjusting its rotation in the X-Y plane until the interferometer indicates no periodic change in X, and then by geometrically aligning the piezoelectric stage by eye.

Misalignment of the specimen axis with the measurement axis will lead to a geometric error proportional to $1 - \cos(\text{misalignment angle})$. This alignment is checked by scanning and measuring the X position of the center of the long vertical fiducial line at the right side of the pattern (see fig. 3) near its top and bottom ends. The angle of rotation of the specimen can be calculated from the positions of these centers and the nominal distance between them. After the specimen has been mounted and aligned by eye, the alignment is checked and readjusted until the program indicates the specimen is properly aligned. The calibration program will not commence taking data unless the misalignment angle is less than ± 0.1 deg. This allows a maximum cosine error of 1.5 ppm, or 0.105 nm on the longest feature on this photomask. If the specimen is tilted (i.e., the specimen stage is rotated about the Y-axis), the leading and trailing edges of the longer patterns will not both be in focus, and this condition will be detected in the measurements.

A significant potential error source is the Abbé error caused by possible offset between the measurement axis and the specimen axis in combination with angular motion of the scanning stage. The Abbé offset in the linewidth measurement system is less than about 2 mm. Comparison of pitch measurements made on this apparatus and on the NIST Line Scale Interferometer (see sec. 4.6) allow compensation for errors of this type. Small random rotations of the scanning stage contribute to the measured random uncertainty.

4.4 Feature Measurement Sequence

A flow chart of the main steps of the feature measurement sequence is given in figure 6. The feature to be measured is first centered in the field of view, then focused and scanned as the optical profile position and intensity data are taken and stored as two one-dimensional arrays. The data are then low-pass filtered to reduce extraneous noise and processed to find the edge locations. Linewidth is then calculated.

Position and intensity data points are correlated by alternately triggering the interferometer and the a-d converter to take one reading each in a software loop while the scanning stage is moving. There may be a few CPU clock cycles delay between the two readings of a data pair, but this delay is very small (corresponding to 2 nm/ms of delay) and is the same at leading and trailing edges; thus, it cancels in the linewidth or pitch calculation.

Image profiles such as those in figure 7 are presented on the computer screen during data acquisition and processing to allow monitoring system operation. After passing several data quality checks, the results are entered into a database for the SRM being calibrated.

A more detailed description of the measurement sequence and system can be found in reference [3].

4.5 Edge Location Determination

Analysis of optical microscope imaging gives the following equation for image intensity at the edge of a line [5]:

$$T_c = R_t (I_o + I_m + 2\sqrt{I_o I_m} \cos \phi), \quad (1)$$

where T_c is the intensity of the light at the threshold point (edge) on the image profile (see fig. 8); I_o is the intensity of the light passing through the not-perfectly-opaque chromium layer; I_m is the intensity of the light passing through the clear areas (beyond the diffraction peaks); and ϕ is the optical phase difference of the light transmitted through these two areas. R_t is a theoretically derived ratio, of approximately 0.25, which varies slightly depending on the coherence factor, viewing slit width, focus, proximity of the next edge, and other imaging conditions. For the conditions of measurement of this SRM in the NIST calibration system, R_t varies from 0.25 to 0.28 (see last paragraph of this section).

Real microscope images often include some flare light (light scattered off the microscope components illuminating the otherwise opaque features on the photomask from above or reaching the image plane by indirect paths). In nonlaser illumination systems, this light is temporally incoherent with respect to the light comprising the diffraction pattern (image profile) and simply adds incoherently (intensity-wise) to each intensity of the image profile.

To a first approximation the intensity of the flare light is not a function of position across a feature. Therefore, the effect of the flare light can be incorporated into eq (1) by simply subtracting its value from each intensity component on the image profile:

$$I_o = I'_o - I_f; \quad I_m = I'_m - I_f; \quad T_c = T'_c - I_f, \quad (2)$$

where the prime designates an observed intensity (including the effects of diffraction, transmission, and flare) and where I_f is the magnitude of the flare light component in the image profile for each feature. Substituting into eq (1) gives:

$$T'_c = R_t [(I'_o - I_f) + (I'_m - I_f) + 2\sqrt{(I'_o - I_f)(I'_m - I_f)} \cos \phi] + I_f. \quad (3)$$

Both ϕ and I_f must be known to evaluate the threshold condition. I_f is feature and background dependent and must be determined for each feature. For this SRM, where the antireflecting-chromium layer can be considered to be homogeneous, the transmittance (Tr) and ϕ can be taken as constants and I_o can be expressed as $(Tr \times I_m)$. Then, considering that $(I_m - I_o)$ equals $(I'_m - I'_o)$ and substituting in eq (2), it can be shown that

$$I_f = [I'_o - (Tr \times I'_m)] / (1 - Tr). \quad (4)$$

The transmittance of the SRM was determined by using the linewidth measuring system to measure the intensity of light passing through the chromium near the center of the large chromium-covered upper-left quadrant of the mask and found to be about 0.18% of the incident intensity.

There is no known simple method for determining ϕ . However, a recent study of image profiles generated by a computer model [5], developed by D. Nyysönen, of the NIST microscope system indicates that linewidth uncertainty from not knowing this phase difference is no greater than 15 nm if $\phi = \pi/2$ is used in determining the threshold intensity. This uncertainty is included in the systematic error budget. The model is based on the theory of partial coherence and allows variation of image formation conditions such as: linewidth; wavelength of incident radiation; transmittance and phase of the object and background illumination; and slit width. Profiles generated by this model agree very closely with profiles generated from the measurement data. Theoretical profiles were generated for lines and spaces 0.50 to 15 μm wide where the transmittance of the "opaque" areas is 0.2% and ϕ ranges from 0 to π . The results of the study also indicate that the relative threshold intensity varied from 25% to 28% of $(I'_m - I'_o)$ over the range of widths simulated, (fig. 9). Therefore, an algorithm for determining linewidth was implemented that assumes a phase difference of $\pi/2$ and iteratively selects the threshold intensity ratio from this model-generated data according to the linewidth of the feature being measured.

4.6 Precision and Accuracy of the SRM Calibration

The SRM certificate reports the measured linewidths and pitches (spacings). The reported values are the average of at least nine repeated measurements. The certified linewidths and pitches have separate uncertainty values because of subtle differences in the way errors affect the measurement of widths and pitches. The uncertainty statement given is based on two contributing factors: precision and accuracy.

Process precision is defined as the variability observed in repeated measurements of a single physical quantity under essentially the same conditions [6] and is intended to describe the reproducibility of the measuring device, the optical microscope, as well as all of the environmental conditions which may affect the measurement [7]. A numerical value for process precision cannot be determined until the measuring system is operating in a state of statistical control and the source of variability is shown to be random in nature and stochastically stationary. When these criteria have been met the process standard deviation quantifies this random error. The value for the process precision on the certificate of calibration includes the variability of the control measurements and the variability of the nine repeated SRM measurements. The details for computing this value are given in the Appendix.

Accuracy is the closeness of agreement between an observed value and the true value [8]. Pitch measurements of several features in a control SRM have been independently calibrated by the Dimensional Metrology Group on the NIST Line Scale Interferometer. Comparison of these measurements with measurements of the same features made on the NIST optical linewidth measurement system provides a scale correction factor to be applied to the SRM calibration measurements. Typically, the scale correction factor is equal or nearly equal to 1.0, indicating there is no significant scale difference between the two measurement systems. This comparison adds a small systematic uncertainty to all measurements (see Appendix, sec. E).

The laser interferometer used to make these dimensional measurements is subject to a sinusoidal nonlinearity along the beam path due to polarization mixing. This leads to a maximum periodic systematic error of ~ 3.5 nm every one-quarter wavelength ($0.16 \mu\text{m}$) for the four beam differential interferometer used here [9]. Since repeat measurements of each photomask feature are made at substantial time intervals, thermal drift in the apparatus insures that these measurements are randomly distributed over this quarter-wavelength period. Thus this error is random, with deviations from the mean wavelength equally likely to be positive or negative, and is included in the calculation of process precision.

The index of refraction of air, and hence the interferometer wavelength, depends on its temperature, pressure, and composition (relative humidity, CO_2 , etc). In this measurement system this can lead to error in two ways: the index error times the interferometer deadpath changes the apparent position in the measurement of each edge, and the index error times the measured length changes the length scale.

The deadpath in this system (the minimum distance between the fixed and moving mirrors) is about 1 cm. The measurement of linewidth or pitch, however, is a differential measurement in that the difference of the positions of the leading and trailing edges is measured by measuring the positions of both edges within a very short time (35 s for the longest pattern on this SRM); the line's width is being measured, not its position. A deadpath error can occur then only if the index of refraction changes in this time interval. Since such changes (caused, for instance, by convective turbulence) are equally likely to be positive as negative, this type of error is random and is included in the measured precision. The maximum magnitude of this deadpath error is 30 nm/°C/min, but since the temperature must decrease after it increases, this error is random and not systematic, relative to the time scale of repeat measurements.

If the temperature is constant but not at the nominal value (20 °C) there will be an index of refraction error of approximately 1 ppm/°C. The worst case error for a 3 °C temperature offset is then 0.21 nm. An atmospheric pressure deviation of 30 mm Hg (4000 Pa) from the nominal 760 mm Hg (101325 Pa) changes the index of refraction by 9 ppm, leading to a 0.63 nm error on the longest feature. Deviation from nominal of the other factors listed above results in similar but much smaller random errors.

In the field of optical submicrometer linewidth metrology, the ideal reference standard with features which have vertical walls and smooth edges does not exist. Instead, real features have erratically varying, nonvertical edge geometries and raggedness along their length [10] (see fig. 10) and the resultant uncertainties of the location of the physical edge. To quantify these systematic uncertainties the feature edge geometry is examined with a scanning electron microscope. As this examination precludes use of the photomask as an SRM, only one sample from each photomask production batch is examined.

The SRM measurements reported represent averages over the effective length (2.6 μm) of the NIST instrument's viewing slit, positioned at the center of the line. Therefore, both the uncertainties of the edge location resulting from nonvertical edge geometry and from raggedness along the length of the line are estimated as averages along the edge of the line.

Typically, the SEM micrographs of the photomask features show that the raggedness along the length of a line is less than 30 nm and has a spatial period of 100 nm or less.³ If the user's measurements of this SRM are also averaged over a length comparable to that of the NIST viewing slit, uncertainties due to edge raggedness become insignificant.

³Occasional isolated flaws have been observed during SEM inspection that are considerably larger than this typical edge raggedness but which are not discernable in the optical microscope at 1600 X magnification and could be present on the photomasks accepted for calibration. If the presence of such flaws in the measurement region should cause degradation of focus sharpness or of measurement precision during the calibration of a photomask, that photomask would be rejected from certification as an SRM. However, it is not known if such flaws would have any noticeable effect on the measurements.

A determination of the uncertainty caused by the nonvertical edge geometry is accomplished by estimating the difference between the edge location at the top surface and the corresponding edge location at the substrate level, as illustrated in figure 11. Estimates are made at uniform intervals along a 1 μm section of the sample as imaged in an oblique view SEM micrograph. The uncertainty is determined as the average of these estimated edge location differences. For width measurements, edge location errors for the right and left edges tend to add; thus the linewidth uncertainty is twice the edge location uncertainty[11].

The linewidth uncertainty value given on the certificate data sheet for rows 1, 2, and 5 includes the uncertainty resulting from the nonideal edge geometry estimated from the SEM micrographs (typically around ± 50 nm), ± 15 nm from not knowing ϕ (see sec. 3.4); and ± 5 nm of less quantitatively defined systematic errors.

Because pitch measurements involve measuring the distance from one location (left edge, right edge, or center) on one feature to the same location on another feature, edge detection errors tend to cancel and are not included in the uncertainty reported for measurements in rows 3, 4, and 6.

The measurement precision is determined for each SRM photomask as described in the Appendix and the pooled 95% confidence levels are reported for the width (rows 1, 2, and 5) and pitch (rows 3, 4, and 6) measurements on the certificate data sheet.

The uncertainty of the width and pitch values reported is a combination of the appropriate systematic uncertainties and measurement precision. A summary of the error components is given in table 1.

The user is advised to examine the edge properties of the production photomasks to be measured. If the quality of the edges of the features on the user's photomasks is significantly inferior to that of this SRM, an additional level of uncertainty should be added to the uncertainty of measurements made on the user's photomasks.

ACKNOWLEDGMENTS

Parts of this document (especially the procedures in sec. 3) are based on the earlier SRM 474 and SRM 475 Handbook prepared by Diana Nyssonen and John Jerke.

The photomicrographs used to examine the edge geometry were provided by the Scanning Electron Microscope Section (Sam Jones, William Keery, and Michael Postek) of the Microelectronics Dimensional Metrology Group.

Many thanks to Robert Larrabee for his guidance and advice and to Beverly Wright for typing the numerous revisions of this document.

REFERENCES

- [1] 1981 Annual Book of ASTM Standards, Part 43, "Standard Practice for Preparing an Optical Microscope for Dimensional Measurements," Designation F 728-81, American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.
- [2] Croarkin, C. and Varner, R.N., "Measurement Assurance for Dimensional Measurements on Integrated Circuit Photomasks," Natl. Bur. Stand. (U.S.) Tech. Note 1164, August 1982.
- [3] Potzick, J., "Automated Calibration of Optical Photomask Linewidth Standards at the National Institute of Standards and Technology," SPIE Vol. 1087, Integrated Circuit Metrology, Inspection, and Process Control, San Jose, CA, February 1989.
- [4] Beers, J., "Length Scale Measurement Procedures at the National Bureau of Standards," Natl. Bur. Stand. (U.S.), NBSIR 87-3625, 1987.
- [5] Nyyssonen, D., "Linewidth Measurement With an Optical Microscope: The Effect of Operating Conditions on the Image Profile," Applied Optics Vol. 16, August 1977, pp. 2223-2230.
- [6] Ku, H.H., "Statistical Concepts in Metrology - With a Postscript on Statistical Graphics," Natl. Bur. Stand. (U.S.), Spec. Publ. 747, August 1988, pp. 12.
- [7] Croarkin, C., "Measurement Assurance Programs Part II: Development and Implementation," Natl. Bur. Stand. (U.S.), Spec. Publ. 676-II, 1985, pp. 23.
- [8] 1986 Annual Book of ASTM Standards, "Statistical Terminology for Statistical Methods," Designation E 456-83a, American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.
- [9] "High Performance Motion Control for Precision Equipment," Hewlett Packard, Co., 1990, p. 34.
- [10] Nyyssonen, D. and Larrabee, R.D., "Submicrometer Linewidth Metrology in the Optical Microscope," J. Res. Natl. Bur. Stand. (U.S.), 92(3), May-June 1987, pp. 189-190.
- [11] Potzick, J., "Practical Photomask Linewidth Measurements," SPIE Vol. 1261-13, Integrated Circuit Metrology, Inspection, and Process Control, San Jose, CA, 1990.

- [12] Bullis, W.M. and Nyyssonen, D., "Optical Linewidth Measurements on Photomasks and Wafers," Chapter 7 in VLSI Electronics: Microstructure Science, Semiconductor Microlithography, Vol. 3, N.G. Einspurch, Editor, pp. 119-126 (Academic Press, New York, NY, 1982).
- [13] Nyyssonen, D., "Linewidth Calibration for Bright-Chromium Photomask," NBSIR 86-3357, Natl. Bur. Stand. (U.S.), May 1986.
- [14] Jerke, J.M., Croarkin, M.C., and Varner, R.N., "Interlaboratory Study on Linewidth Measurement for Antireflective Chromium Photomasks," Natl. Bur. Stand. (U.S.), Spec. Publ. 400-74, 1982.

Table 1. A listing of error sources and their contributions to overall measurement uncertainty. Errors are determined in a worst-case sense; scale factor uncertainties (in ppm) are multiplied by the largest dimension measured, giving scale errors of 0.09 nm (systematic) and 0.40 nm (random) for the largest linewidth (30 μm) and 0.22 nm (systematic) and 0.92 nm (random) for the largest pitch (70 μm). Net random error is derived from the measurements.

SUMMARY OF ERROR COMPONENTS		
<u>Systematic</u>	Linewidth	Pitch
Laser wavelength accuracy	0.1 ppm	0.1 ppm
Axis alignment	3.0 ppm	3.0 ppm
Abbé error ¹	0.0 ppm	0.0 ppm
Substrate thermal expansion ¹	0.0 ppm	0.0 ppm
Refractive index of air ¹	0.0 ppm	0.0 ppm
Chrome edge geometry ²	50 nm	0.0 nm
Chrome transmission magnitude & phase ²	15 nm	0.0 nm
Comparison to Standard meter	2.0 nm	3.0 nm
Other errors	3 nm	0.0 nm
TYPICAL SYSTEMATIC UNCERTAINTY ²	70 nm TOTAL	3.1 nm TOTAL
<u>Random</u>		
Substrate thermal expansion	1.2 ppm	1.2 ppm
Refractive index of air	12 ppm	12 ppm
Deadpath refractive index	6.0 nm	6.0 nm
Polarization mixing	3.5 nm	3.5 nm
Vibration and noise	unknown	unknown
TYPICAL RANDOM UNCERTAINTY ³ (from measurements)	17 nm TOTAL	15 nm TOTAL
¹ These scale factor errors are removed by comparing the linewidth calibration system with the NIST Linescale Interferometer, which measures pitch only, giving traceability to the standard meter (see sec. 4.3, p. 9). ² Typical: depends on photomask batch. ³ One standard deviation for a single measurement. Long-term [6 months, frequent mounting and dismounting] and short-term standard deviations do not differ significantly.		



Figure 1. A view of the overall pattern on SRM 473. The basic measurement pattern is repeated eight times about the center. The horizontal and vertical lines help locate the patterns.

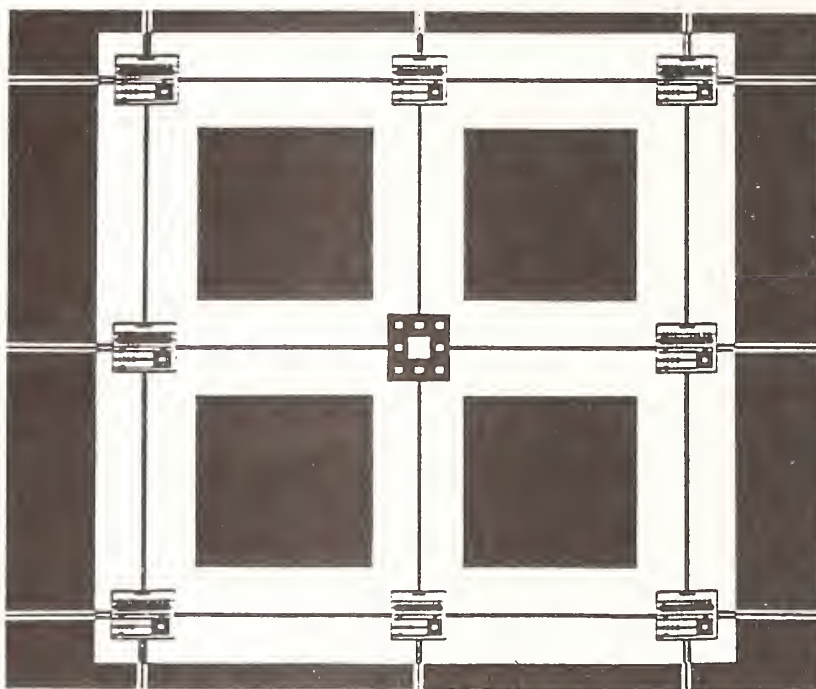


Figure 2. An enlarged view of the center of the SRM. The pattern number given with the serial number on the certificate identifies which basic pattern has been calibrated by NIST. Pattern No. 1 is in the upper left; pattern No. 8 is in the lower right. Pattern identification numbers are included within each basic pattern as shown in figure 3.

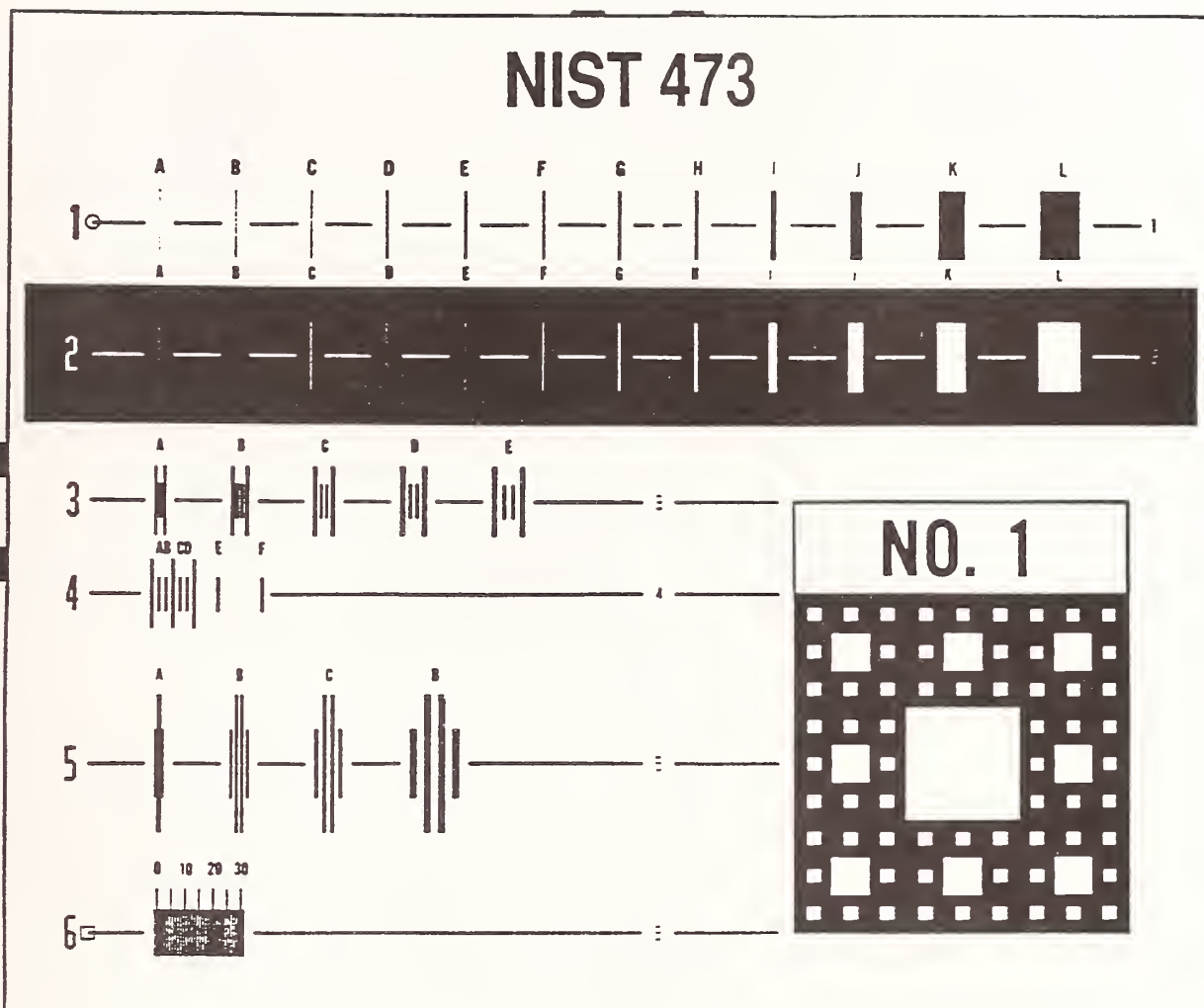


Figure 3. A view of one basic measurement pattern on the SRM. The individual features are located by reference to an alphanumeric code with numbers identifying the row and letters designating the position within the row. The broken horizontal lines mark the central calibrated area of the features. The box surrounding the overall pattern is used to align the pattern on the measurement system. The pattern identification number can be seen in the box above the carpet design in the lower right.

Calibration values are given for: widths of opaque lines in row 1 and clear lines in row 2; center-to-center spacing of the two inner (short) lines of each feature in row 3; center-to-center spacing of line A to lines B through F in row 4; widths of the left inner (long) line and the space to its right of each feature in row 5; and center-to-center spacings of line 0 to lines one through 30 in row 6.

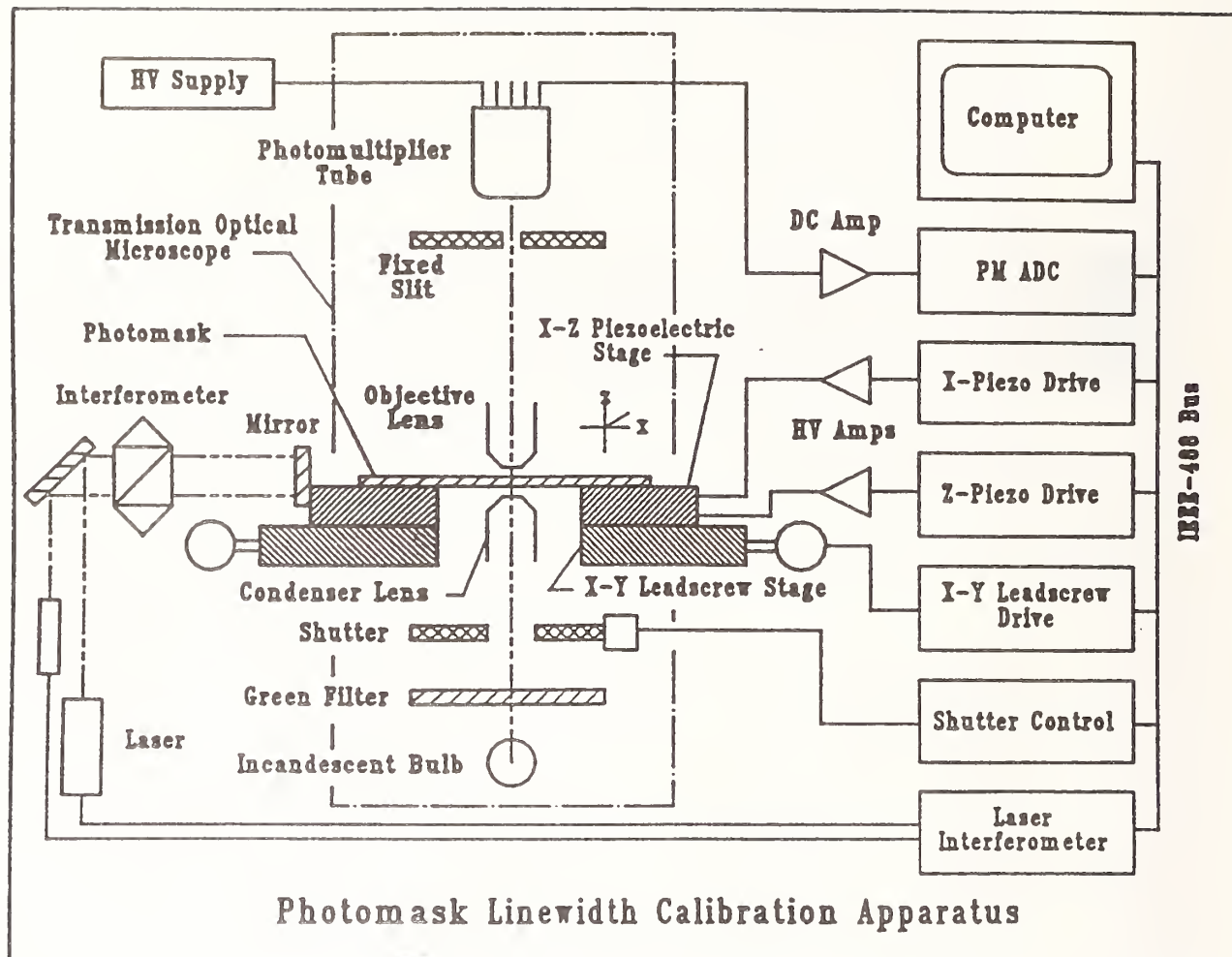


Figure 4. Schematic of the NIST automated optical linewidth calibration system. The photomask is placed on the scanning piezoelectric stage and is illuminated from below with partially coherent light from a filtered incandescent source. The measuring slit remains fixed while the image of the feature being measured is scanned past the slit by moving the photomask. The motion is measured with a laser interferometer and the image intensity at the slit is monitored with a photomultiplier. The digitized and amplified output of the photomultiplier and the interferometer output are connected via the IEEE-488 bus to the computer.

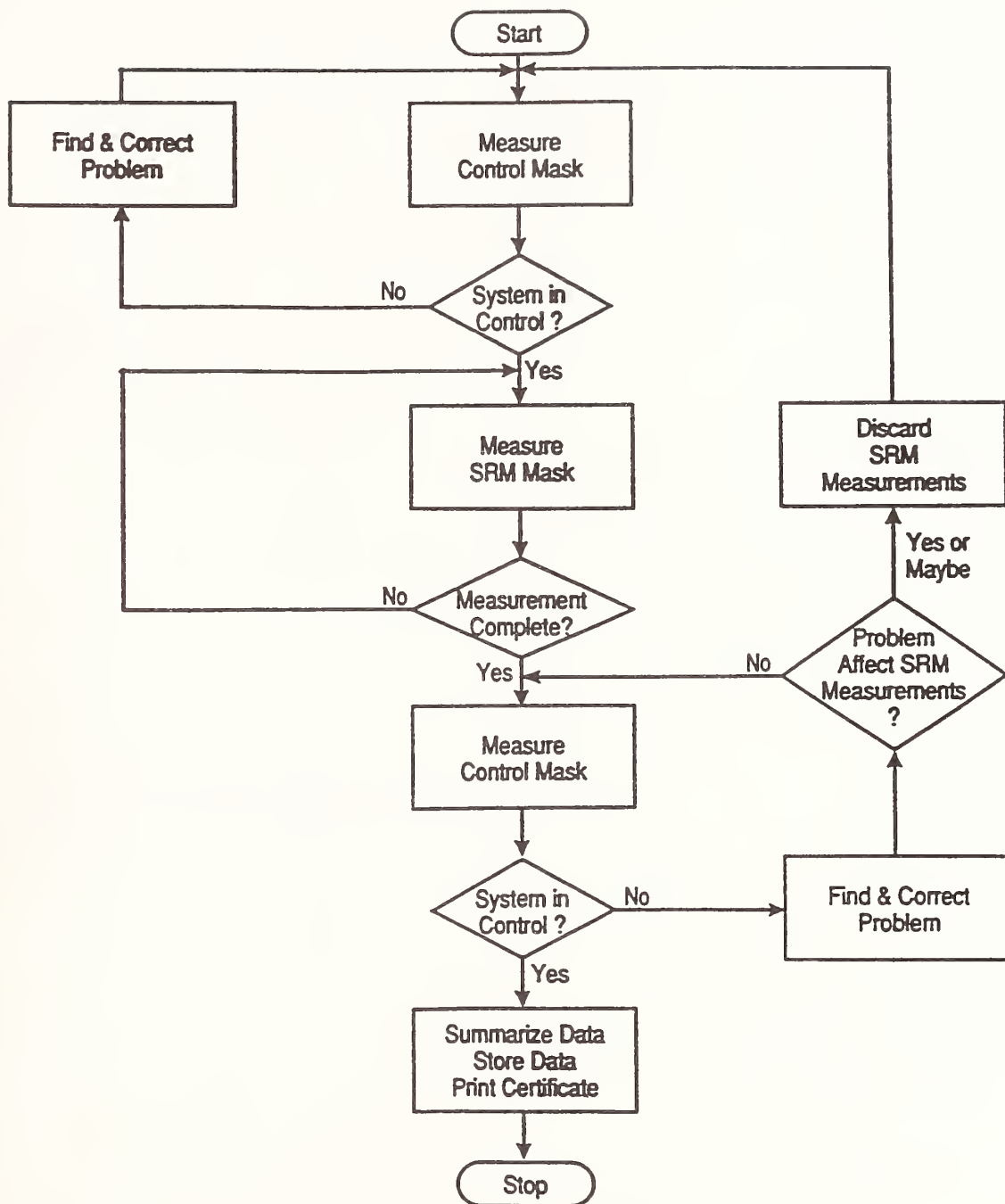


Figure 5. Flow chart outline of the overall calibration procedure for the calibration of SRM photomasks with the NIST optical linewidth measurement system. First, measurements are made on a control photomask and tested statistically to determine if the system is operating properly. Then, the SRM photomask is calibrated and the system operation is checked again by measuring the control photomask. If all tests indicate the system is within statistical control, a calibration certificate can be printed for the SRM photomask.

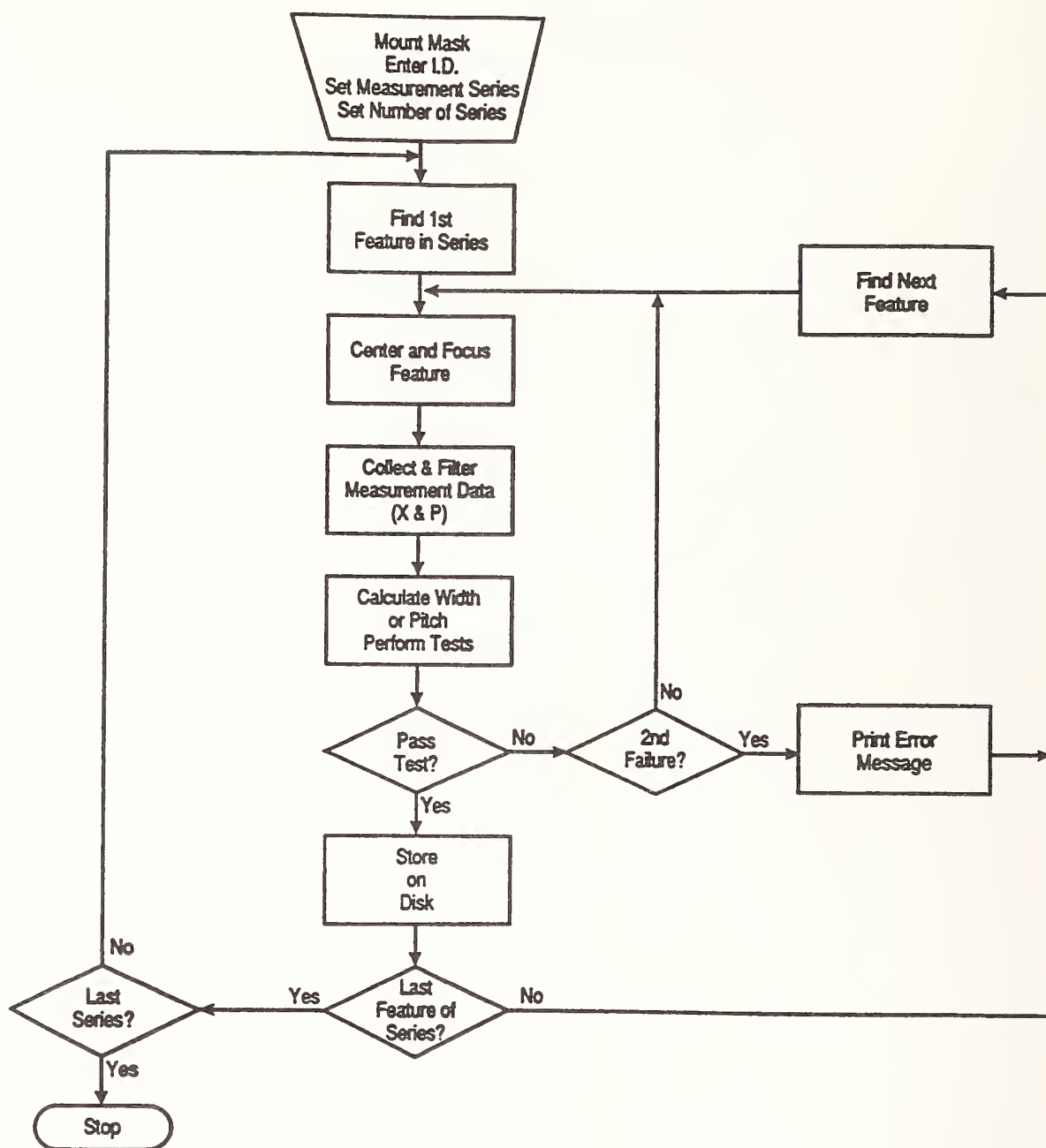
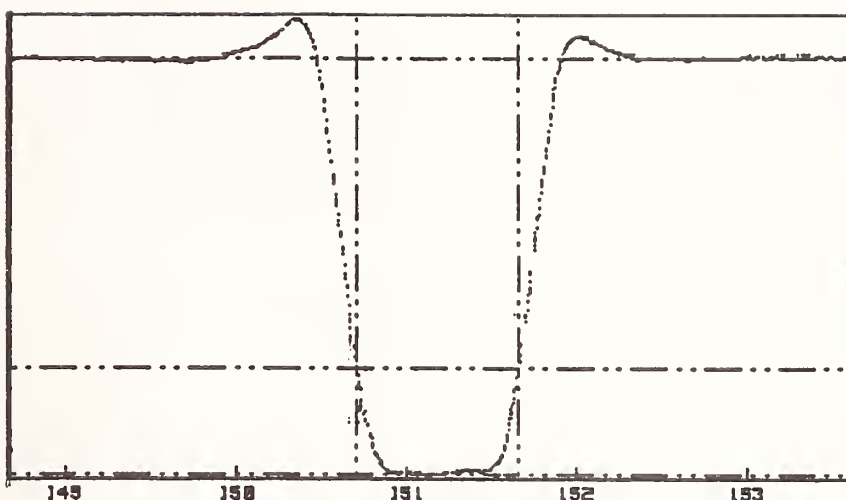
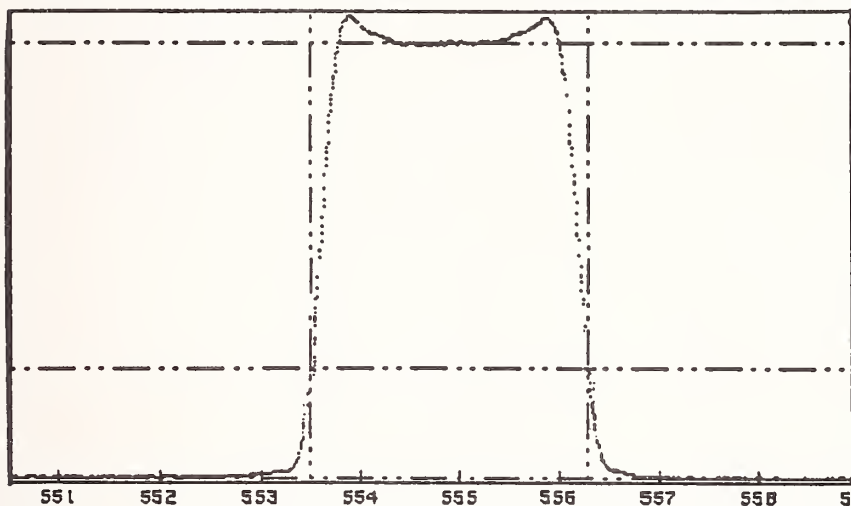


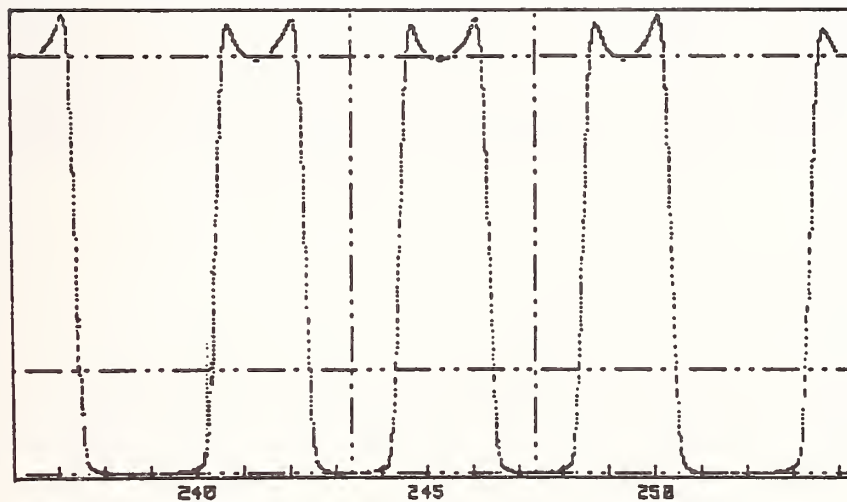
Figure 6. Flow chart of the main steps of the measurement sequence performed by the NIST optical linewidth measurement system. Each feature on the SRM photomask is centered in the microscope field of view, focused, and measured in sequence. The sequence is repeated until each feature has been measured nine times. The optical profile position (X) and intensity (P) are measured and the data are stored as two one-dimensional arrays.



A. Profile typical of an opaque line in row 1. The vertical fiducials mark the positions of the line edges.



B. Profile typical of a clear line (space) in row 2. The vertical fiducials mark the positions of the space edges.



C. Profile typical of a line pair in row 3. The vertical fiducials mark the positions of the line centers. The profile includes the two outer guard lines as well as the line-spacing pair.

Figure 7. Samples of optical profiles (measured light intensity versus position) displayed on the computer screen during the calibration process. The vertical axes are relative light intensity and the horizontal axes are position in micrometers. The horizontal lines are fiducials marking the relative intensity levels of the clear and coated areas and the threshold.

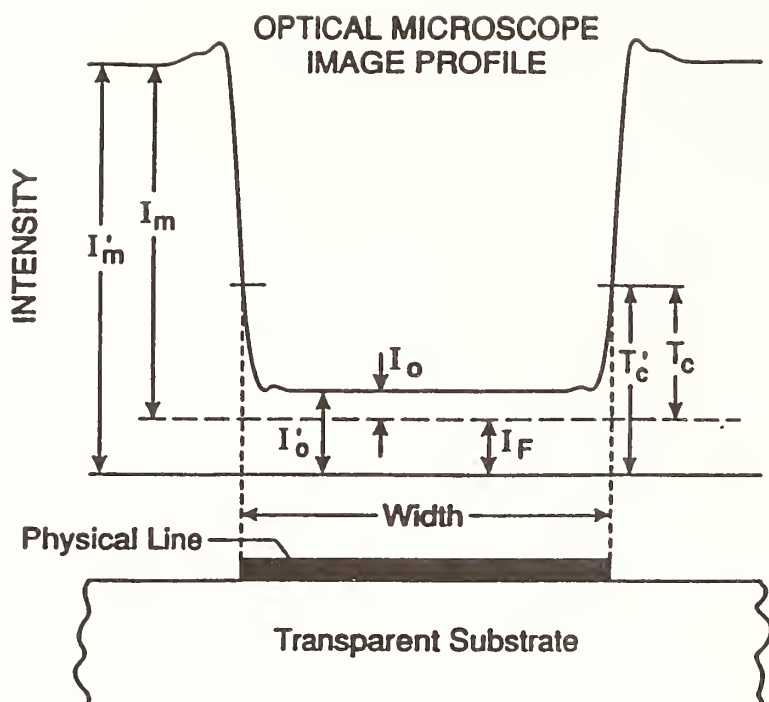


Figure 8. Schematic of the cross section of a vertical-edged chromium line and the corresponding optical profile of its microscope image. I_m is the intensity of the light passing through the clear area; I_o is the intensity of the light passing through the chromium; T_c is the intensity at the physical edge (threshold); I_F is the intensity of the flare light. The prime designates an observed intensity. The vertical axis is optical intensity and the horizontal axis is distance.

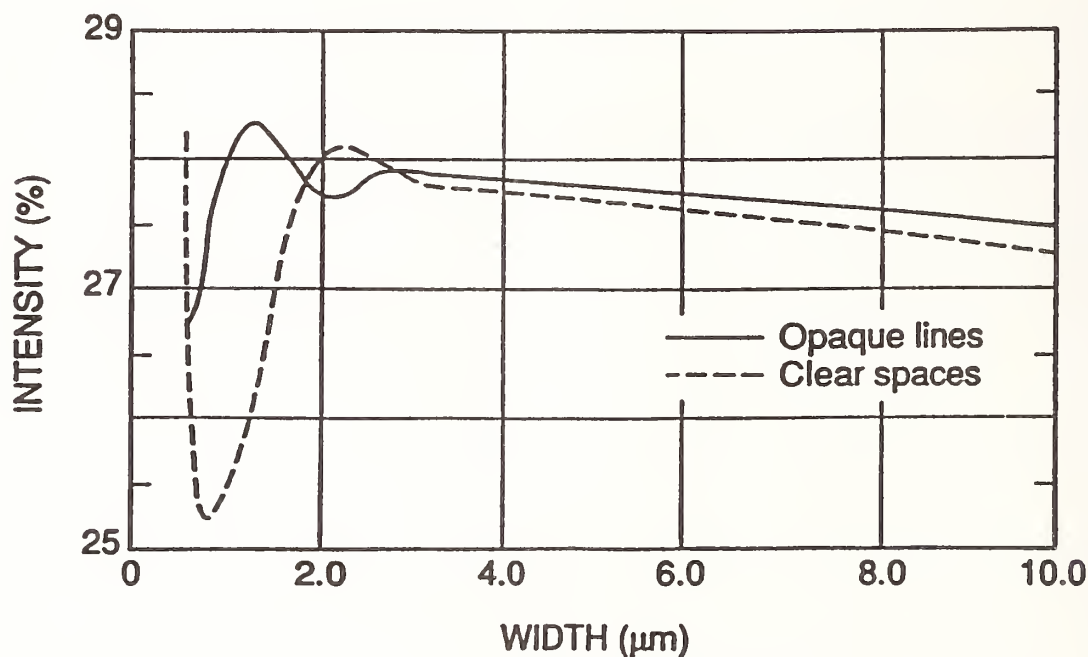


Figure 9. Optical intensity [% of ($I_m - I_o$)] at edge location versus linewidth, from a computer model of the NIST calibration system. Transmittance equals 0.2% and ϕ equals $\pi/2$ radians.



Figure 10. SEM micrographs showing the nonideal nature of line edges on an antireflecting-chromium photomask. The scale is indicated by the row of eleven dots in the lower right of each micrograph. The dots are 20 nm apart. Beam energy was 25 keV.

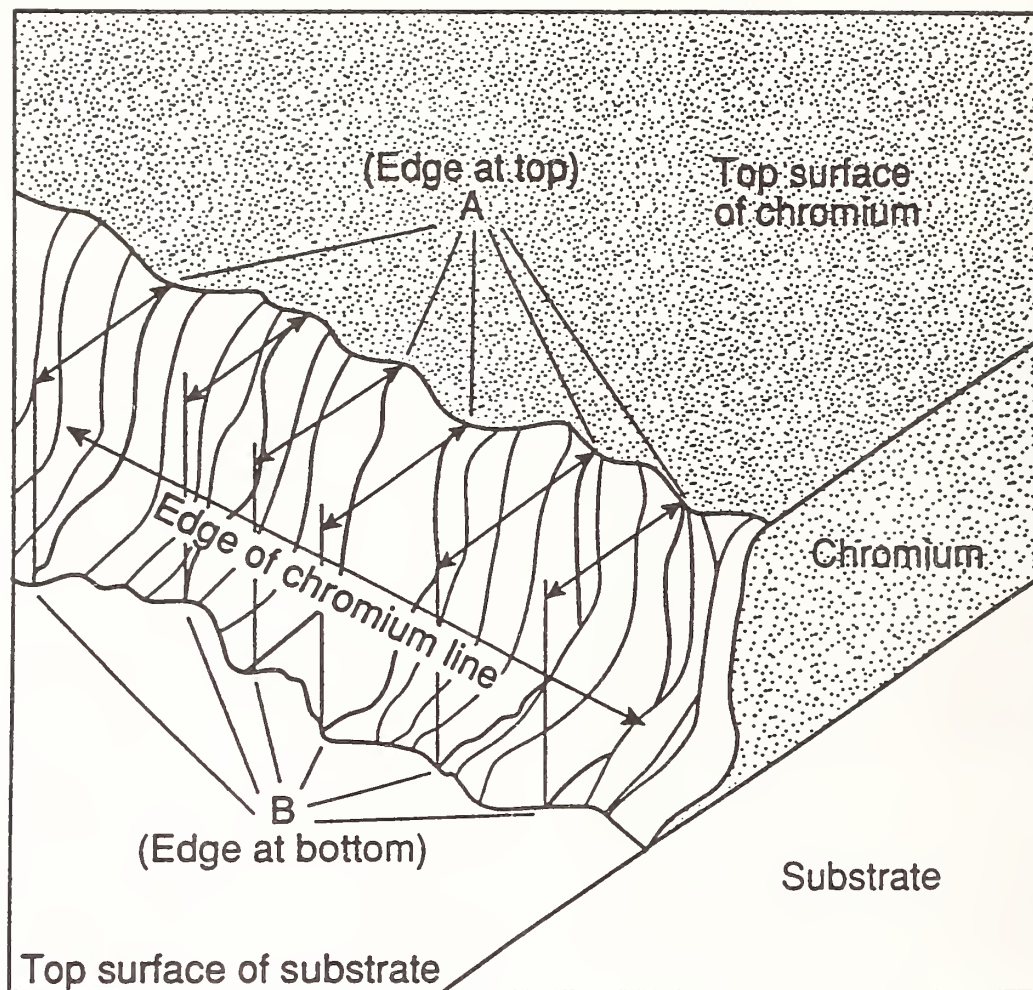


Figure 11. Schematic representation (not to scale) of a line edge as seen in an oblique view SEM micrograph. The uncertainty of linewidth measurements includes the uncertainty of the edge location resulting from non-vertical physical edge profiles. Determination of this uncertainty is accomplished by estimating the difference between the edge location at the top surface (A) and at the substrate level (B). Estimates are made at uniform intervals along a section of a specimen image in an SEM micrograph. The edge uncertainty reported is the average of such estimates made by several individuals using several different micrographs.

Appendix

Process Control for SRM 473 Calibrations

A. Introduction

The procedures used to assure statistical control of the linewidth SRM measurement system are defined. A control photomask with the same characteristics as the SRM photomask is used for measurement process control. Six of the features on the control photomask are measured each time an SRM photomask is calibrated. The six features are: the nominal 0.6 and 5.0 μm lines from row 1; the nominal 1.0 and 20.0 μm lines from row 2; the nominal 6.2 pitch pattern from row 3; and the nominal 2.0 μm line from row 5. These correspond to features 1B, 1I, 2F, 2K, 3E, and 5B as shown on the diagram of a pattern in figure 12. The purpose of the control photomask measurements is to provide a database that can be used to determine whether or not the measurement system is in a state of statistical control. There are several factors which may cause the optical measurement system to be out-of-control. There may be a change in the measurement system or a change in environmental conditions. This document describes the initialization of the database of control measurements, use of the database to determine if the measurement system is in control, and the maintenance of the database over a long period of time.

B. Initialization of Process Parameters

When the measurement system is ready for performing SRM calibrations, a database is initialized. This database consists of at least 15 sets of repeated measurements of the six selected features on the control photomask taken over a period of several weeks [15]. This period is representative of the normal operating mode of the optical measurement system. The six features measured are identified as 1B, 1I, 2F, 2K, 3E, and 5B. These features cover the extremes of the feature sizes and the range of the feature locations on the photomask. The database includes not only the measured linewidth or spacewidth but also other pertinent information such as the date and time of the measurement, feature identification and any other potentially useful information (temperature, scan rate, etc.).

A plot of the repeated measurements for each feature, measured width or pitch versus time, is made to detect any possible anomalies in the measurement system and to verify that the system produces stable measurements whose variability is random in nature. The control database is accepted as being representative of the normal operating environment of the measurement system if no more than 5% of the measurements are suspected outliers (unexplained anomalies). If this is not the case, an effort is made to determine the cause and appropriate adjustments are made to the measurement system. The control database is then reinitialized.

The initial control database is used to estimate the mean vector (accepted mean values for each control feature) and the matrix of covariances between them. These are required elements for the multivariate Hotelling's T^2 test statistic [16]. The details for computing the estimate of the mean vector and the matrix of covariances are given below. The use of this test statistic and updating procedure for this statistic are given in following sections of this document.

From the database of control measurements for features 1B, 1I, 2F 2K, 3E, and 5B, a matrix $[X]$ is constructed, as shown below, of the N initial repeated measurements on the control photomask. Each of the features has the same number of repeated measurements,

$$X_{ij} = \begin{bmatrix} X_{1B,1} & X_{1B,2} & \dots & X_{1B,N} \\ X_{1I,1} & X_{1I,2} & \dots & X_{1I,N} \\ X_{2F,1} & X_{2F,2} & \dots & X_{2F,N} \\ X_{2K,1} & X_{2K,2} & \dots & X_{2K,N} \\ X_{3E,1} & X_{3E,2} & \dots & X_{3E,N} \\ X_{5B,1} & X_{5B,2} & \dots & X_{5B,N} \end{bmatrix} \quad \begin{array}{l} \text{where } i = 1,2,\dots,6 \\ \text{and } j = 1,2,\dots,N. \end{array} \quad (B.1)$$

The average is computed for each of the features based on the N repeated measurements,

$$\begin{aligned} \bar{X}_{1B} &= \sum_{j=1}^N \frac{X_{1B,j}}{N} & \bar{X}_{1I} &= \sum_{j=1}^N \frac{X_{1I,j}}{N} \\ \bar{X}_{2F} &= \sum_{j=1}^N \frac{X_{2F,j}}{N} & \bar{X}_{2K} &= \sum_{j=1}^N \frac{X_{2K,j}}{N} \\ \bar{X}_{3E} &= \sum_{j=1}^N \frac{X_{3E,j}}{N} & \bar{X}_{5B} &= \sum_{j=1}^N \frac{X_{5B,j}}{N}. \end{aligned} \quad (B.2)$$

These values are the elements of the vector of means as denoted below:

$$M = \begin{bmatrix} \bar{X}_{1B} \\ \bar{X}_{1I} \\ \bar{X}_{2F} \\ \bar{X}_{2K} \\ \bar{X}_{3E} \\ \bar{X}_{5B} \end{bmatrix}, \quad (B.3)$$

A matrix is computed of the differences of the measured values minus the mean values,

$$Z_{ij} = X_{ij} - M_i \quad \begin{array}{l} \text{where } i = 1,2,\dots,6 \\ \text{and } j = 1,2,\dots,N, \end{array} \quad (B.4)$$

and the variance-covariance matrix, S , of size 6×6 , of the control database is computed with elements:

$$S_{ij} = \frac{1}{N-1} \sum_{k=1}^N (Z_{ik}) (Z_{jk}) \quad \text{where } i = 1, 2, \dots, 6 \text{ and } j = 1, 2, \dots, 6. \quad (\text{B.5})$$

The inverse of the variance-covariance matrix is computed and is used in conjunction with future control measurements to determine if the measurement system remains in a state of statistical control.

C. Procedures for Process Control

At the beginning of an SRM measurement session the features 1B, 1I, 2F, 2K, 3E, and 5B on the control mask are measured and the multivariate Hotelling's test statistic T^2 is computed as follows:

$$T^2 = \left[\frac{N}{N+1} [Y - M]' S^{-1} [Y - M] \right], \quad (\text{C.1})$$

where Y is a vector of newly determined widths and pitches for the above mentioned features.

The system is in control at a 95% confidence level if

$$\frac{(N-6)}{(N-1)6} T^2 \leq F_{.05}(6, N-6), \quad (\text{C.2})$$

where the $F_{.05}(6, N-6)$ values are found in table 2. The value $(N-6)$ corresponds to ν in table 2.

At the end of the SRM measurement session, the control is remeasured and the test is repeated. If the system is still in control the SRM data are summarized and a certificate of calibration produced. The value of $((N-6)/6(N-1))T^2$ is saved in the control database and the system is ready for the next SRM measurements.

If the test indicates the system is not in control, the data are tagged when they are saved in the control database. The system is then checked to determine the cause of the test failure. A control chart may be used to determine which feature is causing the problem or to see trends in the control data. A control chart for each feature is constructed from the control database as follows:

The mean, \bar{X} , and the standard deviation, $\hat{\sigma}$, for each feature are computed using the N repeated measurements from the control database:

$$\bar{X} = \frac{\sum_{i=1}^N X_i}{N} \quad \text{and} \quad \hat{\sigma} = \sqrt{\frac{\sum_{i=1}^N (X_i - \bar{X})^2}{(N - 1)}}. \quad (\text{C.3})$$

Control limits are computed by using the following equations:

$$\begin{aligned} & \bar{X} \pm \hat{\sigma} t_{.975}(N-1) \text{ for the } 2\sigma \text{ limit} \\ \text{and} & \\ & \bar{X} \pm \hat{\sigma} t_{.995}(N-1) \text{ for the } 3\sigma \text{ limit.} \end{aligned} \quad (\text{C.4})$$

The values for t are found in table 3. The value $(N-1)$ denotes degrees of freedom, df , in table 3.

Figure 13 is an example of a control chart of the initial 70 measurements of feature 1B. Future measurements are added to the chart. The control limits remain the same until the process parameters are updated.

If it is determined that the cause of the failure did not affect the SRM measurements (for example, the control photomask was misaligned), the appropriate adjustments are made and the control photomask is remeasured. If the test then shows the process is in control, the SRM data are summarized, a certificate of calibration produced, and the system is ready for the next SRM measurements.

If it is determined that the cause of the failure may also have affected the SRM measurements (for example, the air-conditioning unit malfunctioned during calibration), the SRM must be re-measured after the problem has been corrected and the test indicates the system is once again in control. Major changes to the measurement system dictate reinitialization of the database.

D. Updating Process Parameters

If the measurement system remains unchanged after collecting a minimum of 30 new (good) sets of control photomask measurements, the process parameters, M , S , and $\hat{\sigma}$ are updated. Equation (B.3) is used to compute M_2 , a vector of estimated means for the recently collected control measurements; eqs (B.4) and (B.5) are used to compute S_2 , the corresponding variance-covariance matrix; and eq (C.3) is used to compute $\hat{\sigma}_2$, a vector of standard deviations for the repeated measurements for each feature. In the updating process, values that have been flagged as out of control are omitted.

Before updating the control database, a comparison is made between the two databases, the old versus the new, to determine whether or not there is a significant difference in terms of the mean vectors and the variance-covariance matrices. The equivalency of variance-covariance matrices is tested as follows:

$$\text{let } \ell = N_1 + N_2, \quad (\text{D.1})$$

where N_1 = number of repeated observations in the control database
and N_2 = number of repeated observations in the new set of control observations.

The new control database will contain both new and old measurements.

Let

$$S = \frac{(N_1 S_1) + (N_2 S_2)}{\ell}, \quad (\text{D.2})$$

where S_1 is the variance-covariance matrix of the current control database
and S_2 is the variance-covariance matrix of additional new control measurements.

Compute the statistic [17]:

$$D = 0.5N_1 \text{ trace } [(S_1 - S)S^{-1}]^2 + 0.5N_2 \text{ trace } [(S_2 - S)S^{-1}]^2; \quad (\text{D.3})$$

and test whether:

$$D \leq \chi_{df}^2(0.05).$$

D is distributed as a chi-square random variable with df (degrees of freedom) = $0.5p(p+1)$ where $p = 6$, the number of features measured. The value of $\chi_{21}^2(0.05)$ is 32.67. If $D \leq 32.67$, then the differences between the old and new covariance matrices can be attributed to random measurement error at the 95% confidence level. However, if the test fails, ($D > 32.67$), this suggests that the process has changed in some manner and the cause needs to be identified and evaluated. If the change is significant, appropriate action must be taken and the control process re-initialized.

If the covariance matrices are statistically the same, the means are compared. To do this, first a pooled covariance matrix is computed:

$$S_p = \frac{[(N_1 - 1)S_1 + (N_2 - 1)S_2]}{\ell - 2}, \quad (\text{D.4})$$

where S_1 and S_2 are defined in (D.2).

Then the statistic is computed:

$$T^2 = \frac{(\ell - p - 1)(N_1 N_2)}{(\ell - 2)p} (\mathbf{M}_1 - \mathbf{M}_2)' S_p^{-1} (\mathbf{M}_1 - \mathbf{M}_2) \quad (\text{D.5})$$

and tested whether:

$$T^2 \leq F_{.05}(p, \ell - p - 1)$$

where N_1 , N_2 and ℓ are defined in (D.1),

\mathbf{M}_1 is the mean vector for the current database,

\mathbf{M}_2 is the mean vector for the newly collected control data,

and $p = 6$, the number of measured features.

T^2 is a random variable with an F-distribution with p degrees of freedom in the numerator and with $\ell - p - 1$ degrees of freedom in the denominator. The $F_{.05}(p, \ell - p - 1)$ value is given in table I. If $T^2 > F_{.05}(p, \ell - p - 1)$, this suggests that there has been a change in the measurement process. The change needs to be identified and appropriate action needs to be taken to reestablish the measurement system and begin the process control anew. However, if $T^2 \leq F_{.05}(p, \ell - p - 1)$ then the differences between the old and new mean vectors can be attributed to measurement error at the 95% confidence level. Since the test for equality of means was only performed if the hypothesis of equal covariance matrices was not rejected, it can be said that there has been no statistically discernable change in the measurement process at the 90% confidence level and the control may be updated to include the new measurements. The covariance matrix is updated as shown in eq (D.4) and the current covariance matrix is:

$$S = S_p. \quad (\text{D.6})$$

The mean vector is updated as shown below:

$$\mathbf{M} = \frac{N_1 \mathbf{M}_1 + N_2 \mathbf{M}_2}{N_1 + N_2}. \quad (\text{D.7})$$

The standard deviation for each feature is updated as follows:

$$\hat{\sigma} = \sqrt{\frac{(N_1 - 1)\hat{\sigma}_1^2 + (N_2 - 1)\hat{\sigma}_2^2}{N_1 + N_2 - 2}}. \quad (\text{D.8})$$

E. Uncertainty Statement for SRM 473

The uncertainties for the certified linewidth and pitch values given in the certificate include small contributions from the measurement precision (random error) and a contribution from the systematic error. The systematic error for both pitch and linewidth values includes a length dependent contribution introduced by correcting the measurements to agree with the NIST Line Scale Interferometer measurements (see sec. 3.5). The systematic error for the linewidth values has a significant contribution (on the order of $\pm 0.05 \mu\text{m}$) resulting from the edge geometry of the features (see sec. 3.5). See table 1 for a detailed summary of error components.

Before determining the total uncertainty for the reported certificate values, it is assumed that all the measurements on the SRM and in the control database have been corrected to compensate for the difference of measurements between the NIST Line Scale Interferometer System and the optical linewidth measurement system. The correction factor is derived by using the model given below and ordinary least squares to estimate α and its variance:

$$X = \alpha Y + \epsilon \quad (\text{E.1})$$

where X represents a measurement from the linewidth measurement system,
 Y represents a measurement from the line scale measurement system,
and ϵ is the random error of measurement.

Then the uncertainties, U_L and U_p , for linewidth and pitch measurements are determined by the equations below:

The variance of each SRM measurement, \hat{s}_j^2 , is

$$\hat{s}_j^2 = \bar{x}_j^2 \frac{\text{var}(\hat{\alpha})}{\hat{\alpha}^2} + \frac{1}{n-1} \sum_{i=1}^n (x_i - \bar{x}_j)^2 \quad (\text{E.2})$$

where \bar{x}_j is the average of the j th feature,
 $\text{var}(\hat{\alpha})$ is the estimated error of the slope,
 $\hat{\alpha}$ is the least squares determination of the slope,
and n is the number of repeated measurements.

The variance of the control measurements, $\hat{\sigma}_k^2$, is

$$\hat{\sigma}_k^2 = \bar{c}_k^2 \frac{\text{var}(\hat{\alpha})}{\hat{\alpha}^2} + CV_k \quad (\text{E.3})$$

where \bar{c}_k is the average of the k th control feature
and CV_k is the k th diagonal element of the variance-covariance matrix for the control data.

Then the pooled variance from the N repeated measurements in the control database and the n repeated measurements of the SRM, \hat{s}_p^2 , is

$$\hat{s}_p^2 = \frac{(N-1) \sum_{k=1}^p \hat{\sigma}_k^2 + (n-1) \sum_{j=1}^q \hat{s}_j^2}{(N-1)p + (n-1)q}. \quad (\text{E.4})$$

The uncertainty for pitch measurements is

$$U_p = \pm(1.96 s_p/\sqrt{n} + \text{systematic pitch uncertainty}) \mu\text{m} \quad (\text{E.5})$$

and the uncertainty for linewidth measurements is

$$U_L = \pm(1.96 s_p/\sqrt{n} + \text{systematic linewidth uncertainty}) \mu\text{m}, \quad (\text{E.6})$$

where s_p is determined by using eqs (E.2), (E.3), and (E.4) for pitch and linewidth measurements on the SRM and in the control database. Typically $s_p = .015 \mu\text{m}$ and $n = 9$ repeat measurements. The factor 1.96 converts the uncertainty to the 95% confidence level.

ACKNOWLEDGMENTS

The authors thank Susannah Schiller for suggesting the statistical tools to assure process control of the linewidth SRM measurement system and for her review of this documentation.

REFERENCES

- [15] Croarkin, C., Measurement Assurance Programs Part II: Development and Implementation, Natl. Bur. Stand. (U.S.) Spec. Publ. 676-II, 1985, p. 35.
- [16] Anderson, T.W., "An Introduction to Multivariate Statistical Analysis," Second ed. John Wiley and Sons, 1984. Chapter 5, pp. 156-190.
- [17] *ibid.*, p. 423.

Table 2. Critical Values of $F_{.05}(6,\nu)$ of the F-Distribution

ν	$F_{.05}(6,\nu)$
10	3.217
12	2.996
14	2.848
16	2.741
18	2.661
20	2.599
22	2.549
24	2.508
26	2.474
28	2.445
30	2.421
32	2.399
34	2.380
36	2.364
38	2.349
40	2.336
42	2.324
44	2.313
46	2.304
48	2.295
50	2.286
52	2.279
54	2.272
56	2.266
58	2.260
60	2.254
62	2.249
64	2.244
66	2.239
68	2.235
70	2.231
72	2.227
74	2.224
76	2.220
78	2.217
80	2.214
82	2.211
84	2.209
86	2.206
88	2.203
90	2.201
92	2.199
94	2.197
96	2.195
98	2.193
100	2.191
102	2.189
104	2.187
106	2.185
108	2.184
110	2.182
112	2.181
114	2.179
116	2.178
118	2.176
120	2.175
∞	2.099

Table 3. Critical Values of $t_{.975}(df)$ and $t_{.995}(df)$ of the Student's t-Distribution

df	$t_{.975}$	$t_{.995}$
10	2.228	3.169
12	2.179	3.055
14	2.145	2.977
16	2.120	2.921
18	2.101	2.878
20	2.086	2.845
22	2.074	2.819
24	2.064	2.797
26	2.056	2.779
28	2.048	2.763
30	2.042	2.750
32	2.037	2.738
34	2.032	2.728
36	2.028	2.719
38	2.024	2.712
40	2.021	2.704
42	2.018	2.698
44	2.015	2.692
46	2.013	2.687
48	2.011	2.682
50	2.009	2.678
52	2.007	2.674
54	2.005	2.670
56	2.003	2.667
58	2.002	2.663
60	2.000	2.660
62	1.999	2.657
64	1.998	2.655
66	1.997	2.652
68	1.995	2.650
70	1.994	2.648
72	1.993	2.646
74	1.993	2.644
76	1.992	2.642
78	1.991	2.640
80	1.990	2.639
82	1.989	2.637
84	1.989	2.636
86	1.988	2.634
88	1.987	2.633
90	1.987	2.632
92	1.986	2.630
94	1.986	2.629
96	1.985	2.628
98	1.984	2.627
100	1.984	2.626
102	1.983	2.625
104	1.983	2.624
106	1.983	2.623
108	1.982	2.622
110	1.982	2.621
112	1.981	2.620
114	1.981	2.620
116	1.981	2.619
118	1.980	2.618
120	1.980	2.617
∞	1.960	2.576

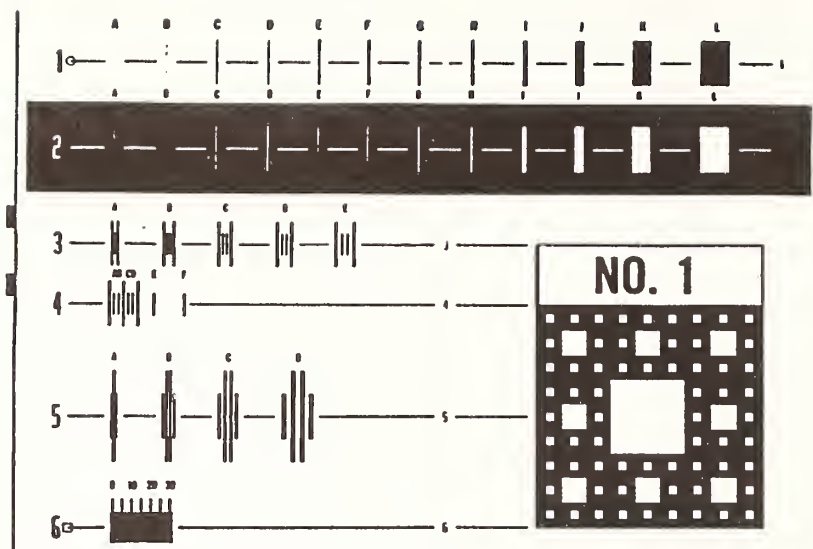


Figure 12. A view of the basic pattern on the SRM 473 control photomask. Features B and I in row 1, F and K in row 2, E in row 3, and B in row 5 are measured before and after each SRM is calibrated on the NIST linewidth measuring system. Feature E in row 3, rows 4 and 6 have been independently calibrated on the NIST Linescale Interferometer by the NIST Dimensional Metrology Group.

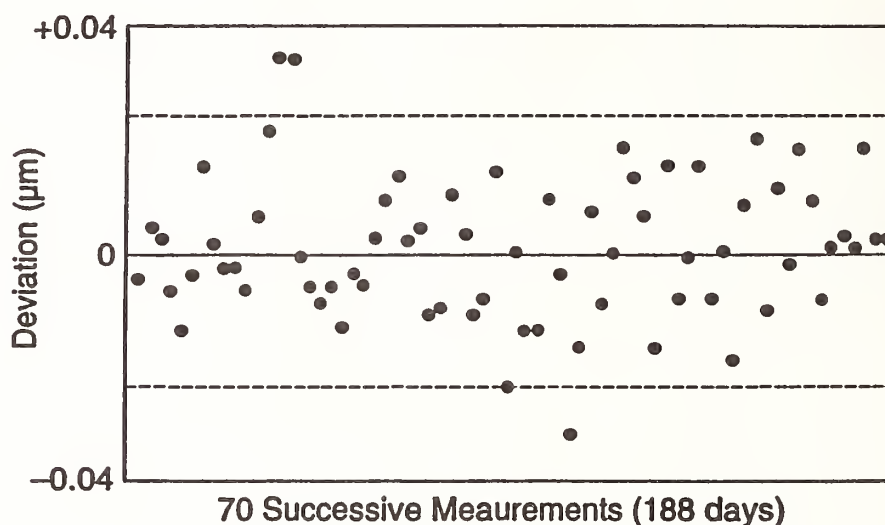


Figure 13. Control chart of feature 1-B. Vertical axis is variation (μm) from the mean (solid line); horizontal axis is successive measurements. The dotted lines mark the limits of the 95% confidence level. Future measurements are added to the chart.

BIBLIOGRAPHIC DATA SHEET

1. PUBLICATION OR REPORT NUMBER NIST/SP-260/119
2. PERFORMING ORGANIZATION REPORT NUMBER
3. PUBLICATION DATE September 1992

4. TITLE AND SUBTITLE Antireflecting-Chromium Linewidth Standard, SRM 473, for Calibration of Optical Microscope Linewidth Measuring Systems

5. AUTHOR(S) Carol F. Vezzetti, Ruth N. Varner, and James E. Potzick

6. PERFORMING ORGANIZATION (IF JOINT OR OTHER THAN NIST, SEE INSTRUCTIONS) U.S. DEPARTMENT OF COMMERCE NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY (MEL) GAITHERSBURG, MD 20899	7. CONTRACT/GRANT NUMBER
	8. TYPE OF REPORT AND PERIOD COVERED Final

9. SPONSORING ORGANIZATION NAME AND COMPLETE ADDRESS (STREET, CITY, STATE, ZIP) Item #6 above.

10. SUPPLEMENTARY NOTES

11. ABSTRACT (A 200-WORD OR LESS FACTUAL SUMMARY OF MOST SIGNIFICANT INFORMATION. IF DOCUMENT INCLUDES A SIGNIFICANT BIBLIOGRAPHY OR LITERATURE SURVEY, MENTION IT HERE.)

The precise and accurate measurement of feature dimensions on photomasks, such as those used in the production of integrated circuits, becomes increasingly difficult as the dimensions approach the wavelength of the light used to make the measurement. The undesirable effects of optical diffraction obscure the location of the feature edges. Raggedness and nonvertical walls along the edges add to the uncertainty of the measurement.

Standard Reference Material SRM 473 was developed for use in calibrating optical microscopes for measuring linewidths in the range of 0.5 to 30 μm on antireflecting-chromium photomasks. The SRM is described, and instructions on its use and precautions concerning its care and handling are given.

The NIST linewidth measuring system and the procedures used to calibrate the SRM are discussed. The algorithm used for determining the line edge location incorporates a threshold criterion derived from analysis of microscope image profiles. The profiles are predicted by computer modeling based on the theory of partial coherence. The performance of this system is monitored by measuring line features on a control photomask before and after calibrating each SRM.

12. KEY WORDS (6 TO 12 ENTRIES; ALPHABETICAL ORDER; CAPITALIZE ONLY PROPER NAMES; AND SEPARATE KEY WORDS BY SEMICOLONS) accuracy; antireflecting-chromium; calibration; control charts; critical dimensions; integrated circuits; linewidth measurement; optical microscope; photomask; precision; semiconductor industry; standard reference material; statistical process control; threshold; uncertainty
--

13. AVAILABILITY	14. NUMBER OF PRINTED PAGES
<input checked="" type="checkbox"/> UNLIMITED FOR OFFICIAL DISTRIBUTION. DO NOT RELEASE TO NATIONAL TECHNICAL INFORMATION SERVICE (NTIS).	48
<input type="checkbox"/> ORDER FROM SUPERINTENDENT OF DOCUMENTS, U.S. GOVERNMENT PRINTING OFFICE, WASHINGTON, DC 20402.	15. PRICE
<input type="checkbox"/> ORDER FROM NATIONAL TECHNICAL INFORMATION SERVICE (NTIS), SPRINGFIELD, VA 22161.	

NIST Technical Publications

Periodical

Journal of Research of the National Institute of Standards and Technology—Reports NIST research and development in those disciplines of the physical and engineering sciences in which the Institute is active. These include physics, chemistry, engineering, mathematics, and computer sciences. Papers cover a broad range of subjects, with major emphasis on measurement methodology and the basic technology underlying standardization. Also included from time to time are survey articles on topics closely related to the Institute's technical and scientific programs. Issued six times a year.

Nonperiodicals

Monographs—Major contributions to the technical literature on various subjects related to the Institute's scientific and technical activities.

Handbooks—Recommended codes of engineering and industrial practice (including safety codes) developed in cooperation with interested industries, professional organizations, and regulatory bodies.

Special Publications—Include proceedings of conferences sponsored by NIST, NIST annual reports, and other special publications appropriate to this grouping such as wall charts, pocket cards, and bibliographies.

Applied Mathematics Series—Mathematical tables, manuals, and studies of special interest to physicists, engineers, chemists, biologists, mathematicians, computer programmers, and others engaged in scientific and technical work.

National Standard Reference Data Series—Provides quantitative data on the physical and chemical properties of materials, compiled from the world's literature and critically evaluated. Developed under a worldwide program coordinated by NIST under the authority of the National Standard Data Act (Public Law 90-396). NOTE: The Journal of Physical and Chemical Reference Data (JPCRD) is published bi-monthly for NIST by the American Chemical Society (ACS) and the American Institute of Physics (AIP). Subscriptions, reprints, and supplements are available from ACS, 1155 Sixteenth St., NW., Washington, DC 20056.

Building Science Series—Disseminates technical information developed at the Institute on building materials, components, systems, and whole structures. The series presents research results, test methods, and performance criteria related to the structural and environmental functions and the durability and safety characteristics of building elements and systems.

Technical Notes—Studies or reports which are complete in themselves but restrictive in their treatment of a subject. Analogous to monographs but not so comprehensive in scope or definitive in treatment of the subject area. Often serve as a vehicle for final reports of work performed at NIST under the sponsorship of other government agencies.

Voluntary Product Standards—Developed under procedures published by the Department of Commerce in Part 10, Title 15, of the Code of Federal Regulations. The standards establish nationally recognized requirements for products, and provide all concerned interests with a basis for common understanding of the characteristics of the products. NIST administers this program as a supplement to the activities of the private sector standardizing organizations.

Consumer Information Series—Practical information, based on NIST research and experience, covering areas of interest to the consumer. Easily understandable language and illustrations provide useful background knowledge for shopping in today's technological marketplace.

Order the above NIST publications from: Superintendent of Documents, Government Printing Office, Washington, DC 20402.

Order the following NIST publications—FIPS and NISTIRs—from the National Technical Information Service, Springfield, VA 22161.

Federal Information Processing Standards Publications (FIPS PUB)—Publications in this series collectively constitute the Federal Information Processing Standards Register. The Register serves as the official source of information in the Federal Government regarding standards issued by NIST pursuant to the Federal Property and Administrative Services Act of 1949 as amended, Public Law 89-306 (79 Stat. 1127), and as implemented by Executive Order 11717 (38 FR 12315, dated May 11, 1973) and Part 6 of Title 15 CFR (Code of Federal Regulations).

NIST Interagency Reports (NISTIR)—A special series of interim or final reports on work performed by NIST for outside sponsors (both government and non-government). In general, initial distribution is handled by the sponsor; public distribution is by the National Technical Information Service, Springfield, VA 22161, in paper copy or microfiche form.

U.S. Department of Commerce
National Institute of Standards and Technology
Gaithersburg, MD 20899

Official Business
Penalty for Private Use \$300